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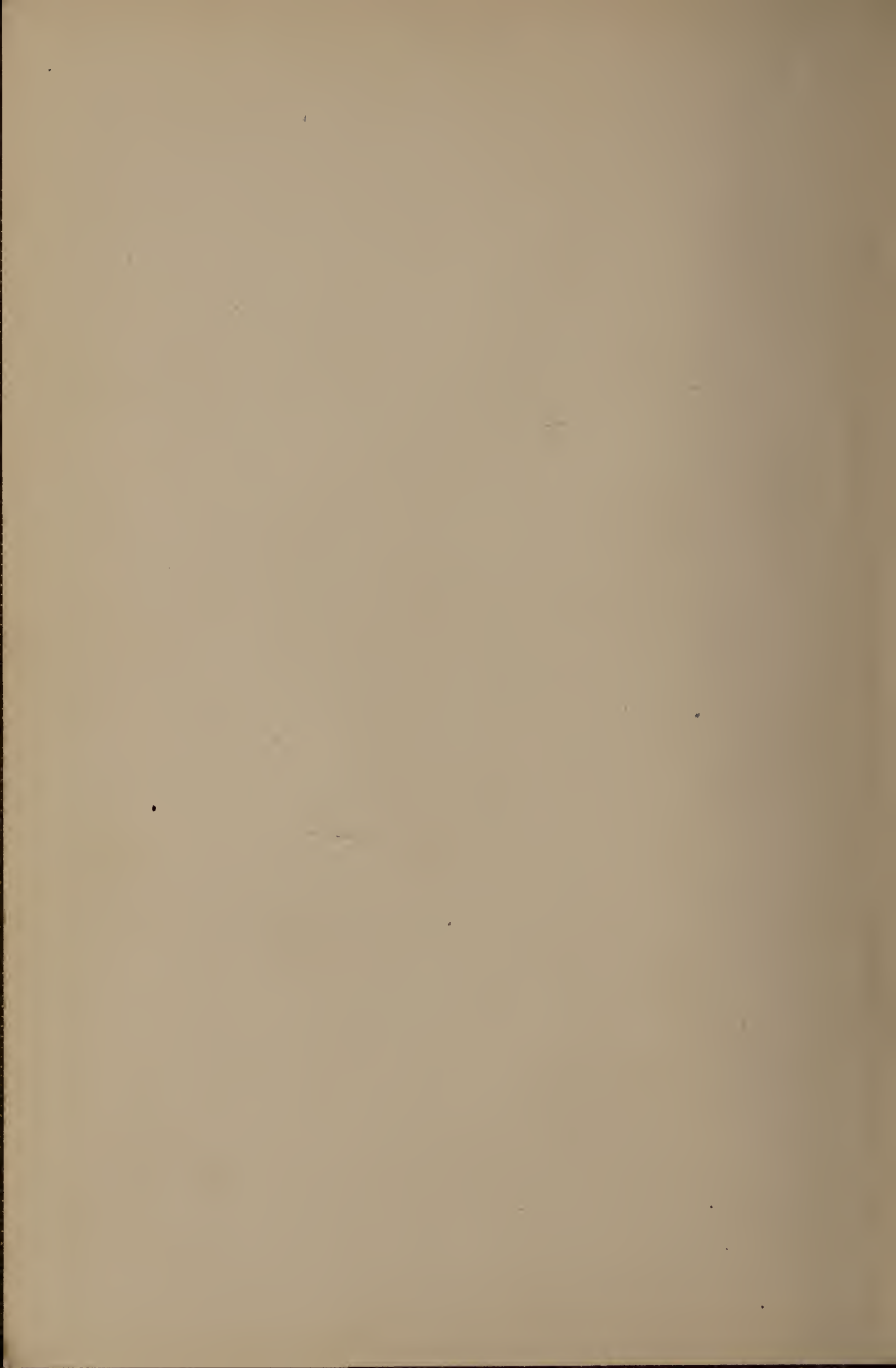


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SCIENCE OF THE METALS

HERBERT R. MOODY





CHEMISTRY OF THE METALS

LABORATORY EXPERIMENTS

BY

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Companion Volume to Estabrooke's
REACTIONS OF CATIONS AND ANIONS



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PREFACE

THE following collection of experiments is the result of several years' experience in teaching under varying conditions.

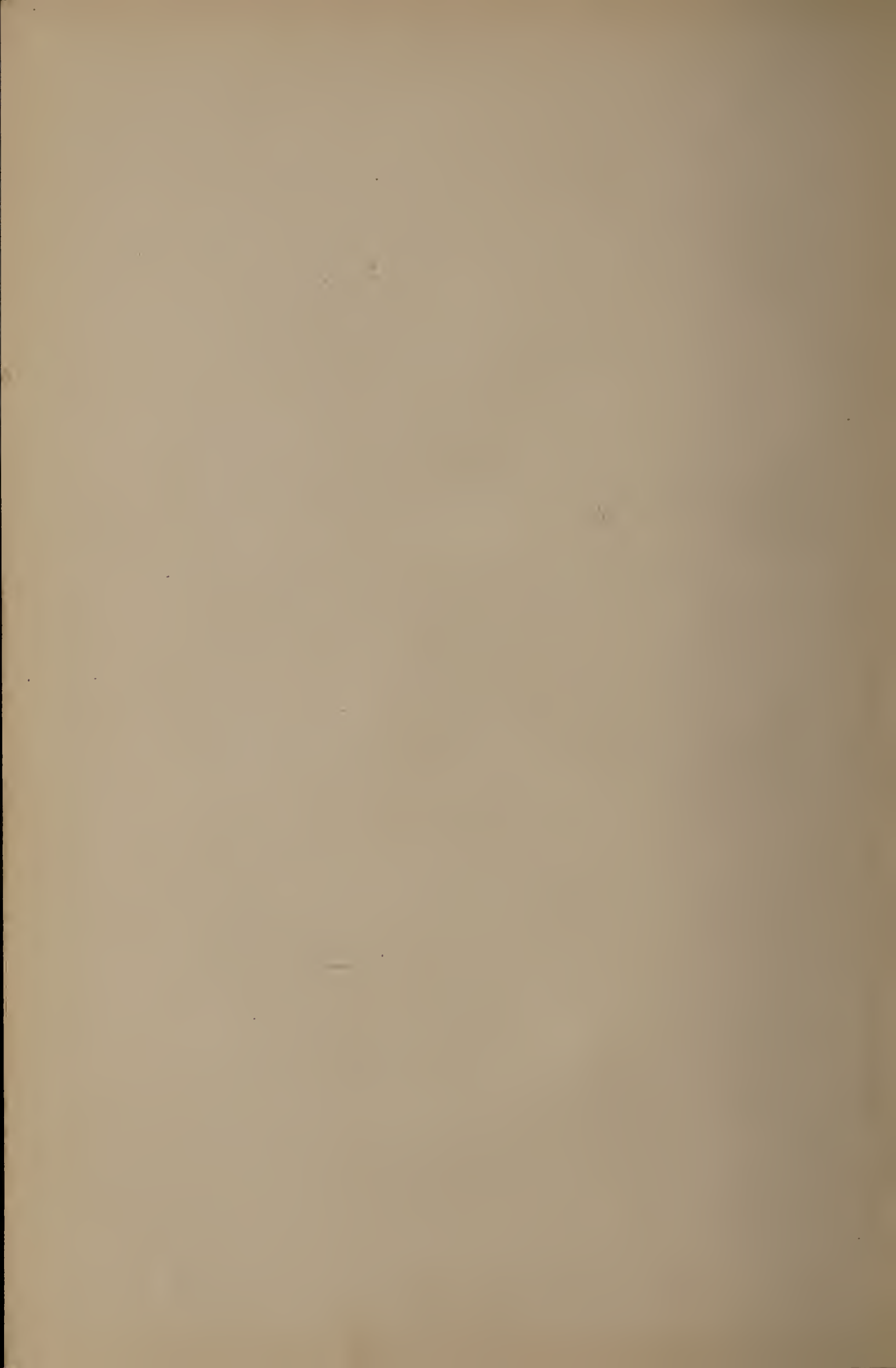
The course is laid out for a half year's work preparatory to qualitative analysis. If it is desired to do in *one term* such preliminary work *together with* elementary qualitative analysis, only those experiments numbered in bold face type need be performed.

The author makes acknowledgment to Professor Lewis M. Norton for some of the experiments in this collection

TO THE STUDENT

Your notes should consist of answers to the following questions:

1. What substances did you use?
2. What did you do?
3. What did you observe? Note whether a precipitate forms, and, if so, whether it is soluble in an excess of the reagent. Note the color of the precipitate and, if it re-dissolves, record the color of the resulting solution.
4. What do you conclude? Write all reactions.



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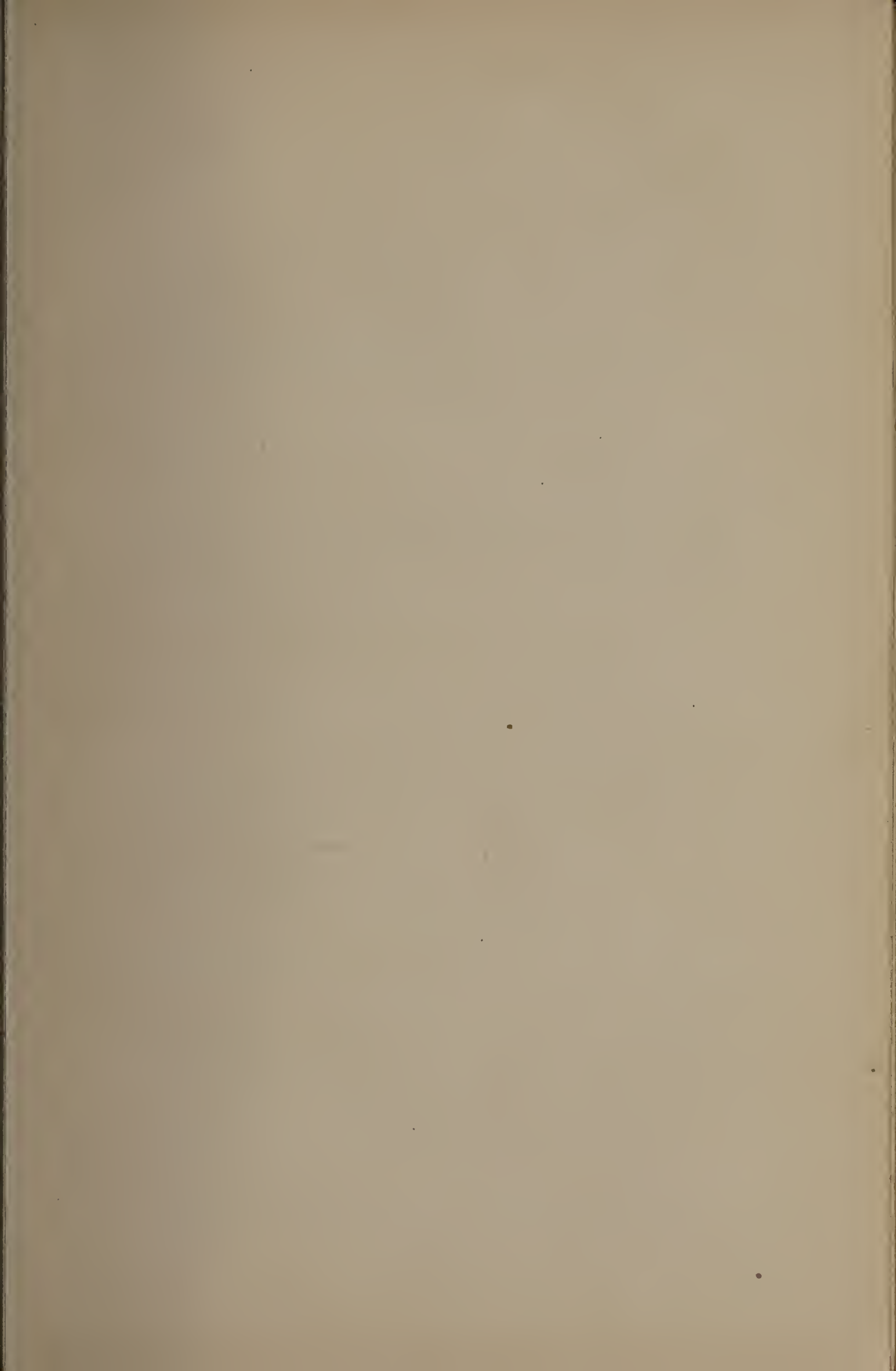
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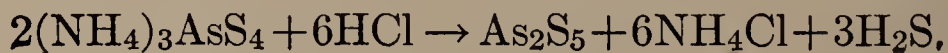
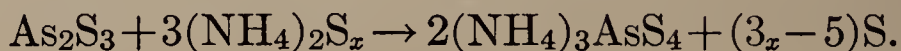


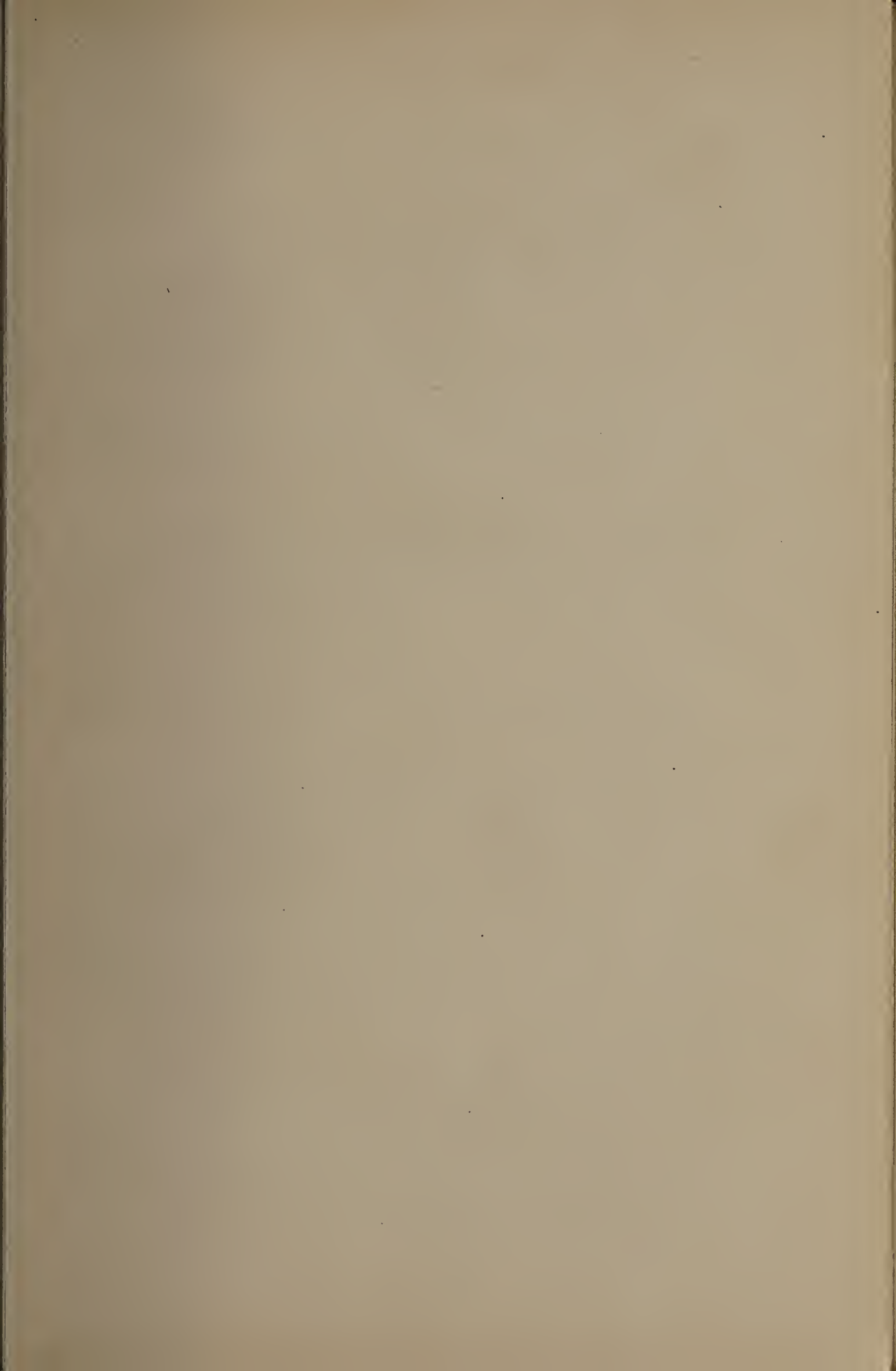
PRELIMINARY EXPERIMENTS

TYPES OF CHEMICAL REACTIONS

Analyze the following reactions and decide whether they are analytical, synthetical, or metathetical. Also explain any divergencies from the action that would naturally be expected.

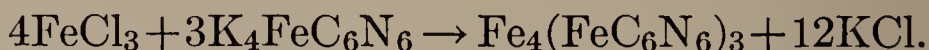
1. To 1 cc. of Na_2CO_3 solution add dilute HCl .
2. Add dilute HNO_3 to metallic copper.
3. Cover metallic copper with concentrated H_2SO_4 and test for gas before and after warming.
4. Mix 2 grams of NH_4Cl with 2 grams of $\text{Ca}(\text{OH})_2$ and test for gas before and after warming.
5. Take 5 cc. of CuSO_4 solution in each of three test-tubes. To the first add an equal volume of NaCl solution; to the second, a few cubic centimeters of BaCl_2 solution. Compare the three tubes.
6. To 5 cc. of $\text{Na}_2\text{S}_2\text{O}_3$ solution add dilute HCl . To a second 5 cc. add $\text{HC}_2\text{H}_3\text{O}_2$.
7. Shake 5 grams of CaSO_4 in hot water, filter, and add $(\text{NH}_4)_2\text{CO}_3$ solution to the filtrate.
8. Take 5 cc. of a solution of As_2O_3 in HCl and add 5 cc. of water. Add $(\text{NH}_4)_2\text{S}_x$, drop by drop, finally in large excess, then boil. Now acidify with HCl :





9. Repeat Experiment 8, using CdCl_2 instead of As_2O_3 .

10. Take 3 cc. of a solution of FeCl_3 in each of two test-tubes. To one add $\text{K}_4\text{FeC}_6\text{N}_6$; to the other add KSCN :



11. Add 3 drops of KSCN solution to 10 cc. of water; then add 1 drop of $\text{Fe}(\text{NO}_3)_3$ solution. Now add AgNO_3 solution, drop by drop, until there is a change of color. *Explain.*

12. To 5 cc. of CuSO_4 solution add a few drops of NH_4OH ; then add the NH_4OH in excess.

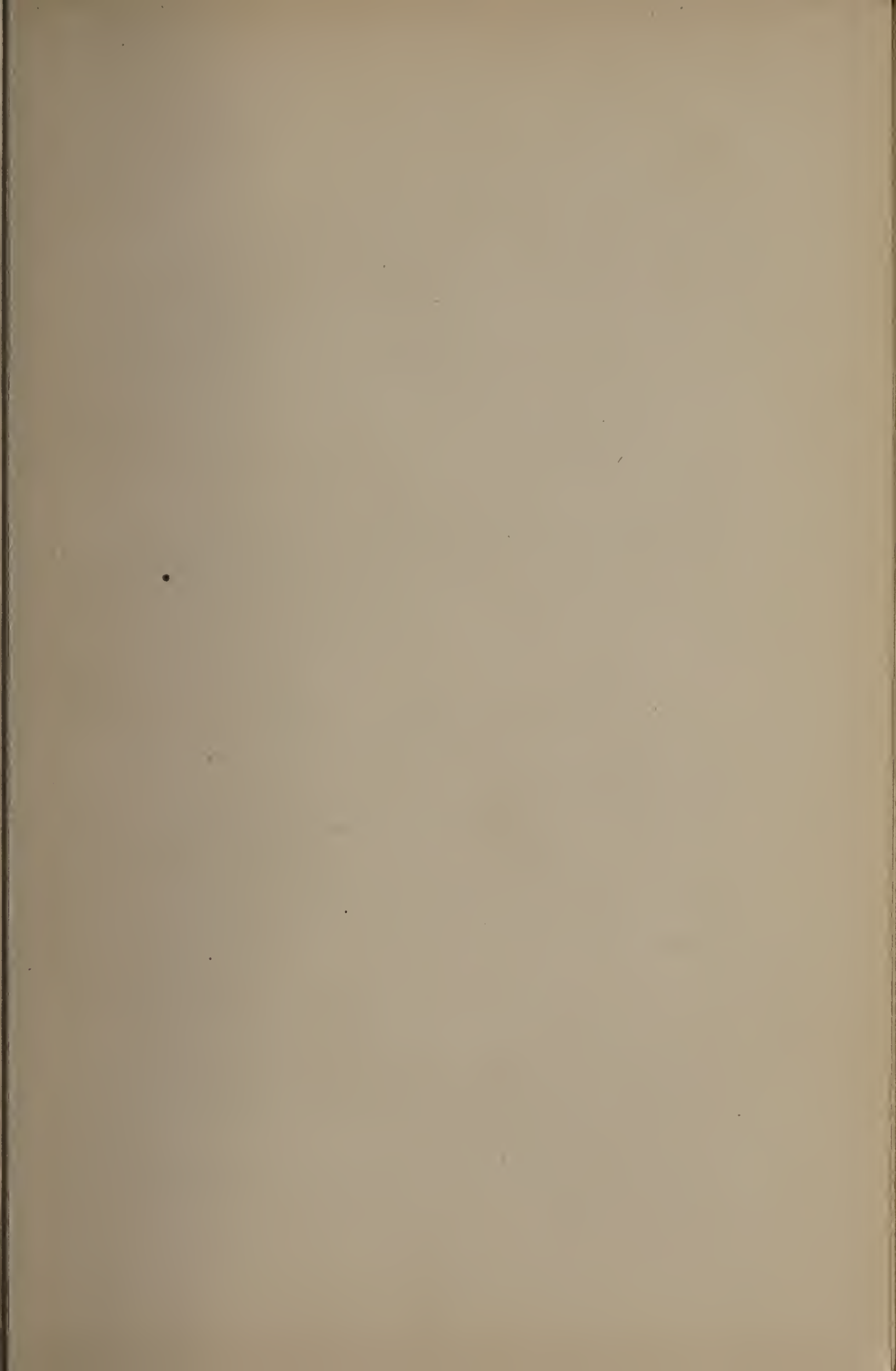
13. Pass CO_2 gas into a solution of Na_4SiO_4 .

SOLUBILITY

Note. Use distilled water. Grind all salts. Note the temperature of the water.

14. Weigh 3 portions of 1 gram each of Na_2SO_4 , CaSO_4 , and PbSO_4 . Take three test-tubes and place 10 cc. of water in each. Into one of the tubes pour one portion of Na_2SO_4 and shake; if this dissolves, add the second, and, if the second dissolves, add the third. Repeat the process with the CaSO_4 and PbSO_4 . If the first portion fails to dissolve entirely, ascertain whether any dissolves by filtering and evaporating a few drops of the filtrate on a clean watch glass.

15. Weigh four portions of 1 gram each of powdered $\text{Ba}(\text{NO}_3)_2$. Place 10 cc. of water in a test-tube, add one portion, and shake. Note the result. Warm slowly, and, as often as the salt is entirely dissolved, add a new portion of 1 gram. Finally bring the liquid to boiling. What does this experiment show?



16. [The salts *must* all be powdered!] Take five test-tubes and place in each 10 cc. of water. Into a sixth test-tube put 12 or 15 grams of potassium carbonate (K_2CO_3). Weigh the tube and contents and record the weight. From this test-tube pour successive, small portions of K_2CO_3 into one of the test-tubes containing water as long as it will dissolve. Again weigh the test-tube and contents. The loss in weight will be the weight of the K_2CO_3 dissolved in the water. Calculate the number of parts of the salt which have dissolved in 100 parts of water. In like manner test the solubility of KNO_3 , K_2SO_4 , $SrSO_4$, and $BaSO_4$, starting with 5 grams of the first, 3 grams of the second, and 1 gram each of the third and fourth. Write the results of the experiment in the form of a table. (See Solubility Tables on page 97.)

USE OF DIFFERENT SOLVENTS

17. Compare the solubility of bone ash in water, dilute HCl, and dilute H_2SO_4 .

18. (**Note.** Use *one small fragment* of solid iodine in each test). Compare the solubility of iodine in water, ethanol, and carbon bisulfide. Use no heat.

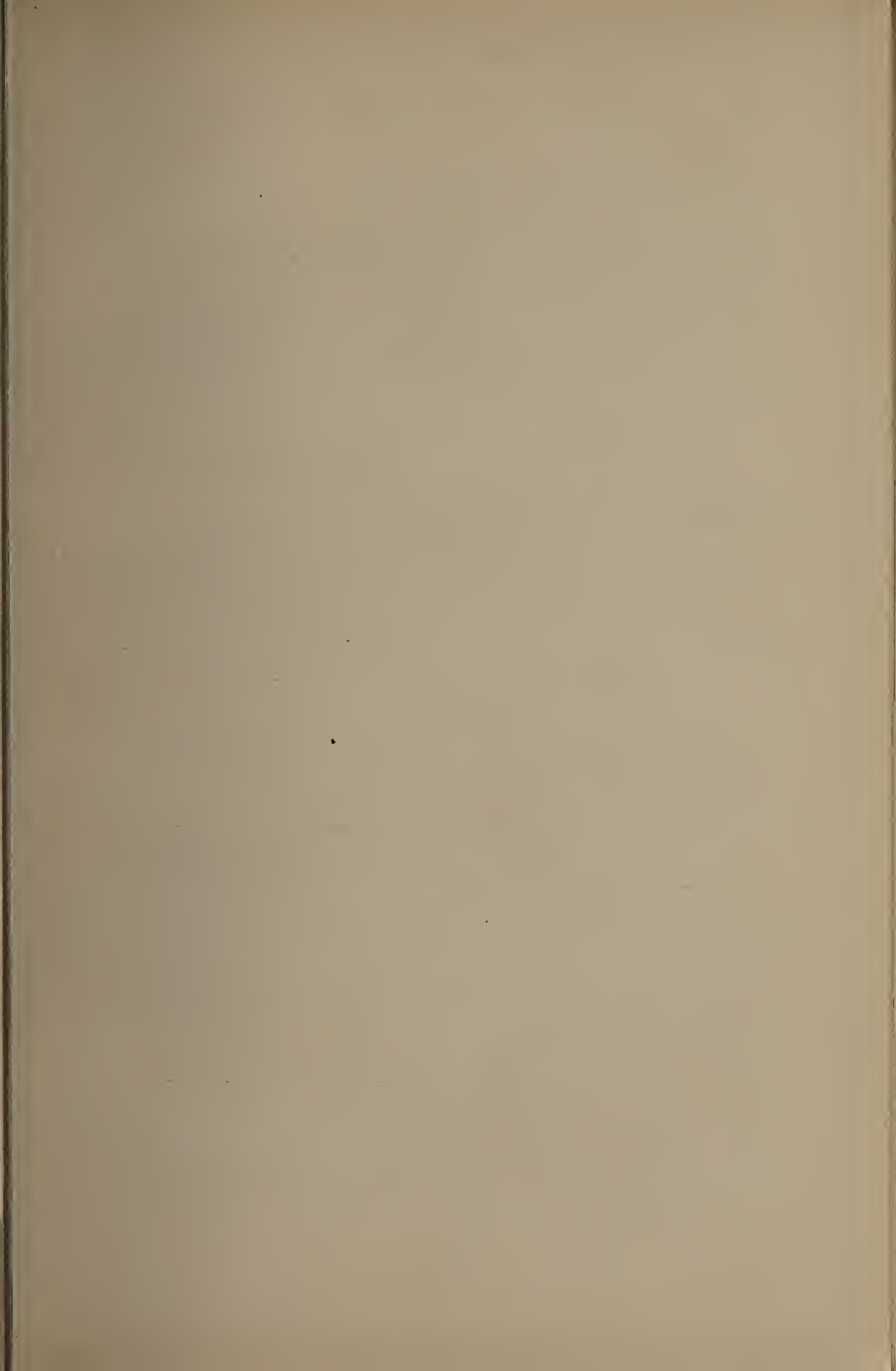
19. Compare the solubility of NaCl in water, strong HCl, and ethanol.

SOLUBILITY IN MIXTURES

20. Attempt to dissolve 1 gram of $BaCl_2$ in 3 cc. of strong HCl. Finally add 10 cc. of water.

21. Dissolve 2 grams of Na_2SO_4 in 4 cc. of water and add an equal volume of ethanol.

22. Dissolve 0.5 gram of iodine in 2 cc. of ethanol, then add 5 cc. of water,



NEUTRALIZATION OF THE SOLVENT

23. Dissolve 2 grams of $\text{Ca}_3(\text{PO}_4)_2$ in warm, dilute HCl, then add NH_4OH to alkaline reaction. Repeat with barium oxalate.

24. Dissolve some freshly precipitated AgCl ($\text{AgNO}_3 + \text{HCl}$) in NH_4OH , then add HNO_3 to the solution to acid reaction.

ABSORPTION AND DEVELOPMENT OF HEAT

25. Note the temperature of 25 cc. of strong (*commercial*) HCl. Add 20 grams of Glauber's salt to the acid, stir, and note the lowest temperature.

26. Note the temperature of 10 cc. of water. Add 10 grams of KOH to the water, stir, and note the highest temperature.

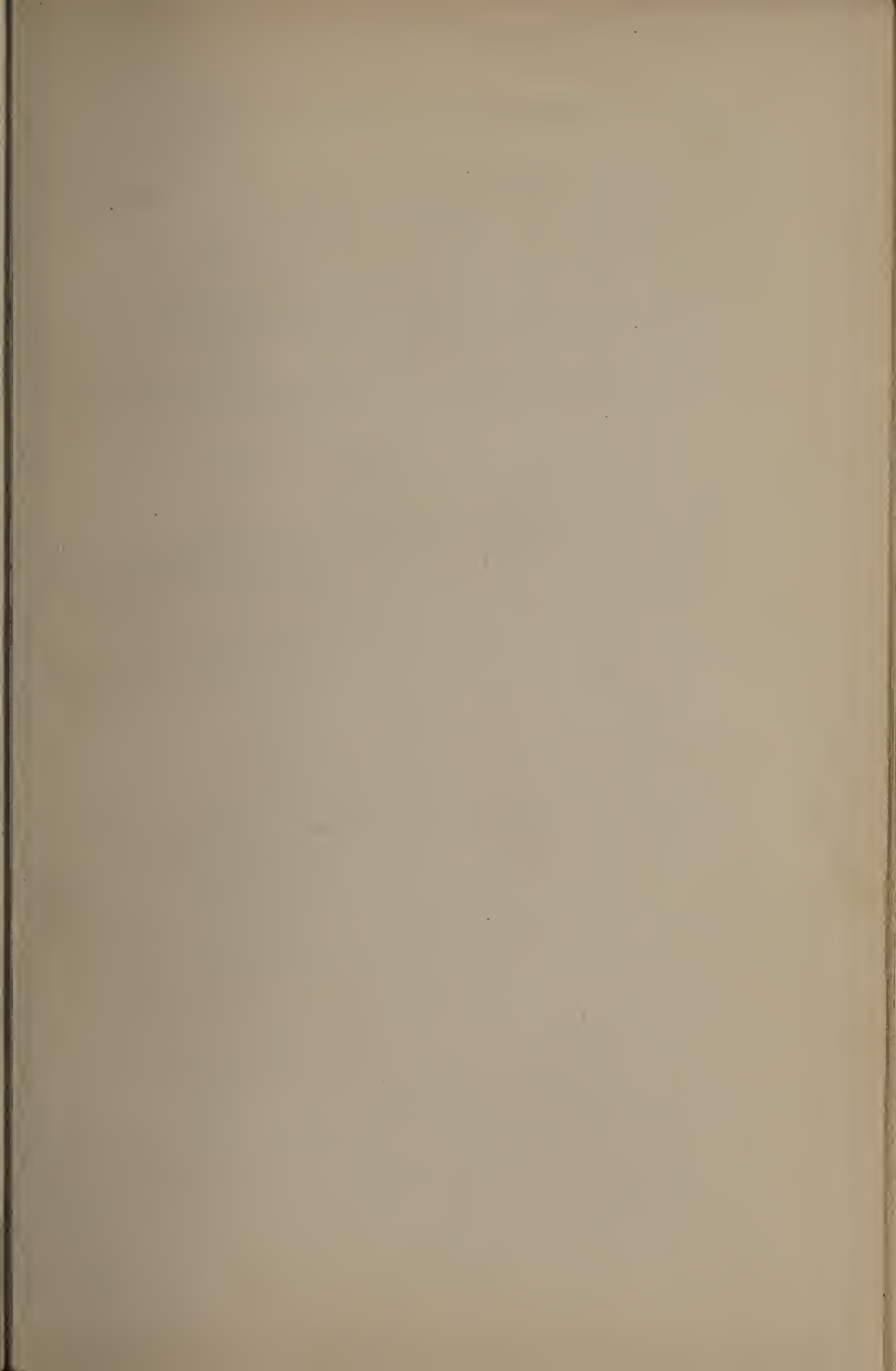
SATURATION

27. Heat together 10 grams of alum and 8 cc. of water and allow to cool. Pour off the clear liquid, boil it for a few moments, and allow it to cool again.

28. Place 10 grams of sodium acetate in a test-tube, add 10 cc. of water, and warm until the sodium acetate has entirely dissolved. Place a little cotton-wool in the mouth of the tube, set it aside, and allow it to become perfectly cool, without moving. The liquid should be clear. Now drop into the liquid a small crystal of sodium acetate and observe the effect.

SOLUTION OF LIQUIDS

29. Test the solubility of the following liquids in water: ethanol, ether, olive oil, glycerine, and carbon bisulfid. Proceed in each case as follows: Take 5 cc. of water in a



clean test-tube and pour 1 cc. of the liquid to be tested upon the water in the tube. Shake several times, and then observe the depth of the liquid layer (if any) above or below the water.

Caution. *Ether and carbon bisulfid are very inflammable and very volatile, and must not be brought anywhere near a flame.*

30. Test the solubility of benzol in water and in ethanol.

PHYSICAL AND CHEMICAL SOLUTION

31. Place 5 cc. of ethanol in a test-tube, add 1 cc. of HCl, and shake. Drop a small piece of fused potassium carbonate into the tube. Now place 5 cc. of water in a test-tube and add 1 cc. of HCl. Drop a small piece of fused potassium carbonate into this tube. *Explain.*

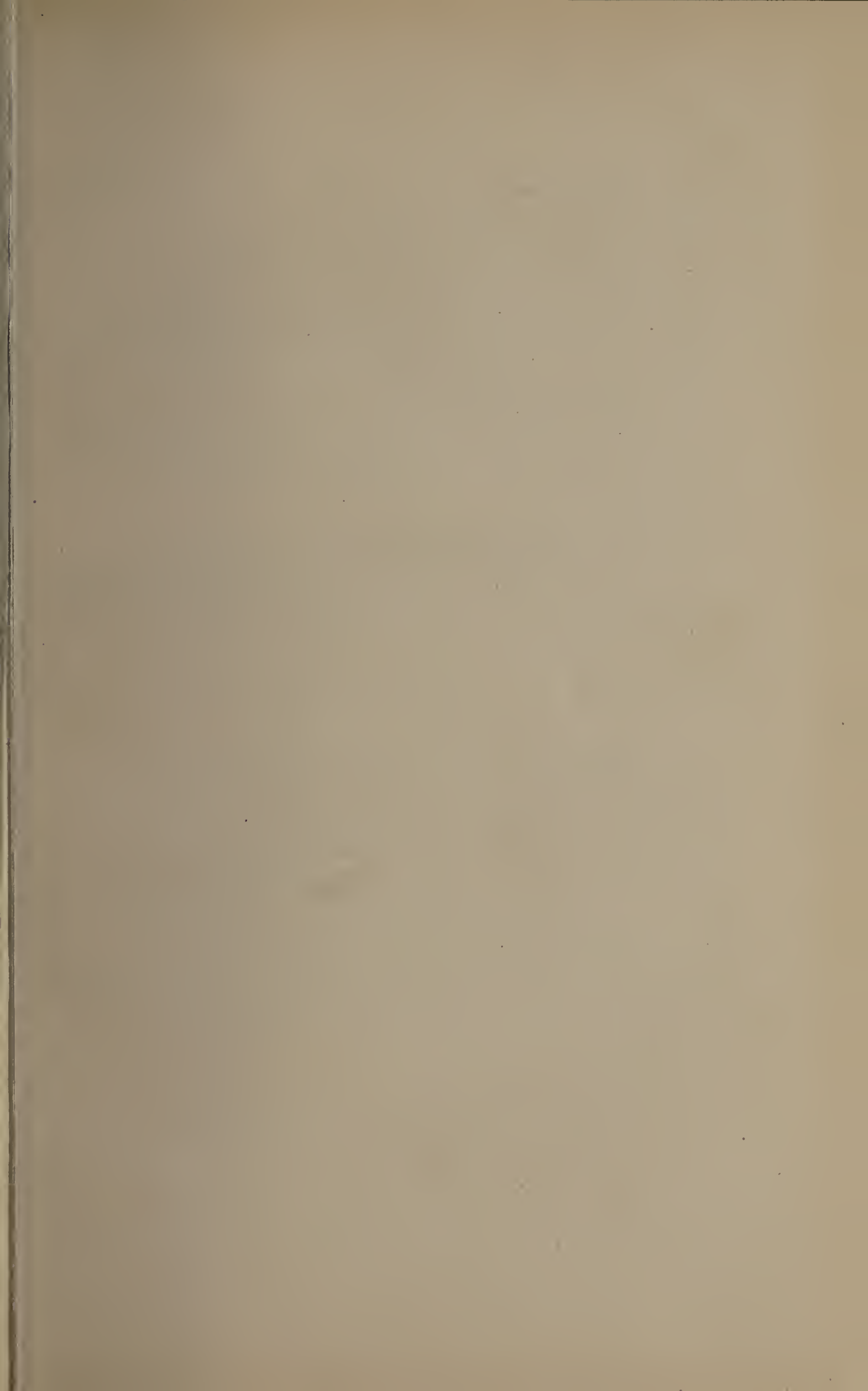
32. Repeat Experiment 31, using CaCO_3 instead of K_2CO_3 .

CRYSTALLIZATION

33. **Crystallization by Solution.** Put about 0.5 gram of PbCl_2 into a test-tube containing 10 cc. of water. Boil for a minute or two, and then filter quickly, receiving the filtrate in a clean test-tube which is kept warm by being immersed in a beaker of hot water. When the liquid has filtered through, remove the test-tube from the hot water and allow it to cool.

34. **Crystallization by Sublimation.** Heat gently a small amount of benzoic acid in a small evaporating dish covered with a watch glass.

35. **Crystallization by Precipitation.** To a few cubic centimeters of a concentrated solution of NaCl add 5 cc. of ethanol.



36. **Crystallization by Solution and Evaporation.** Place upon small watch-glasses 1 cc. each of solutions of potassium nitrate, potassium chlorate, potassium chromate, mercuric chlorid, and sodium acetate. Allow the glasses to stand in your desk until the next laboratory period. Then observe carefully with the lens the forms of the crystals obtained.

37. **Water of Crystallization (hydration.)** Grind together in a mortar 4 grams of crystallized sodium sulfate (*Glauber's salt*) and 2 grams of potassium carbonate. *Explain the result.*

38. Heat native gypsum in a small hard glass tube.

39. Heat a weighed amount of *powdered* native gypsum in a porcelain crucible until a constant weight is obtained. Calculate the percentage of water of crystallization.

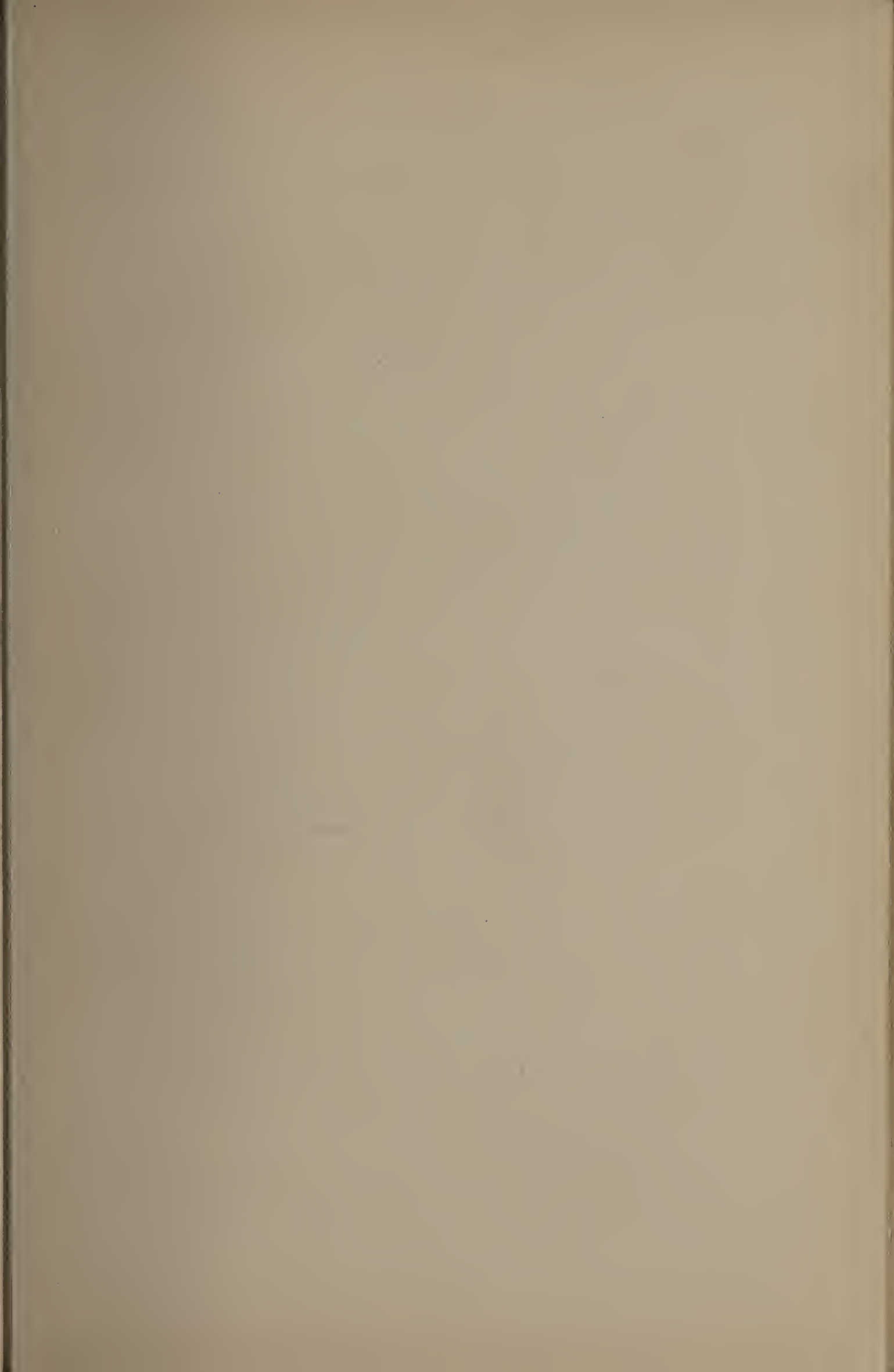
NEUTRALIZATION

40. Measure from a burette a definite quantity of dilute HCl (about 10 cc.), add a few drops of litmus solution, and exactly neutralize the acid with dilute NaOH, drawn from another burette. Repeat, starting with 20 cc. of HCl. Repeat again, starting with 30 cc. of HCl. Put these results in the form of a proportion.

41. Repeat Exp. No. 40, using KOH and HNO_3 .

42. Repeat Exp. No. 40, using NH_4OH and $\text{HC}_2\text{H}_3\text{O}_2$.

43. Repeat Exp. No. 40, using NH_4OH and HCl.



REACTIONS OF THE METALS

SILVER

44. Mix 3 cc. each of $\text{Cu}(\text{NO}_3)_2$ and AgNO_3 solutions, dilute with 10 cc. of water, and insert a strip of metallic copper. Wash off the deposit from the copper, collect it on a filter, and wash. Dissolve this deposit in dilute HNO_3 , add dilute HCl , filter, and to the filtrate add NH_4OH to alkaline reaction.

45. To 5 cc. of AgNO_3 solution add sufficient NH_4OH to make the solution alkaline, then add 3 grams of milk-sugar and 5 cc. of KOH . Warm the mixture gently.

46. Precipitate some AgCl by adding dilute HCl to 5 cc. of AgNO_3 solution. Wash the precipitate on the filter, partially dry it, mix with four times its bulk of dry Na_2CO_3 , and fuse on charcoal. Show your result.



In each of the following experiments use 5 cc. of AgNO_3 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

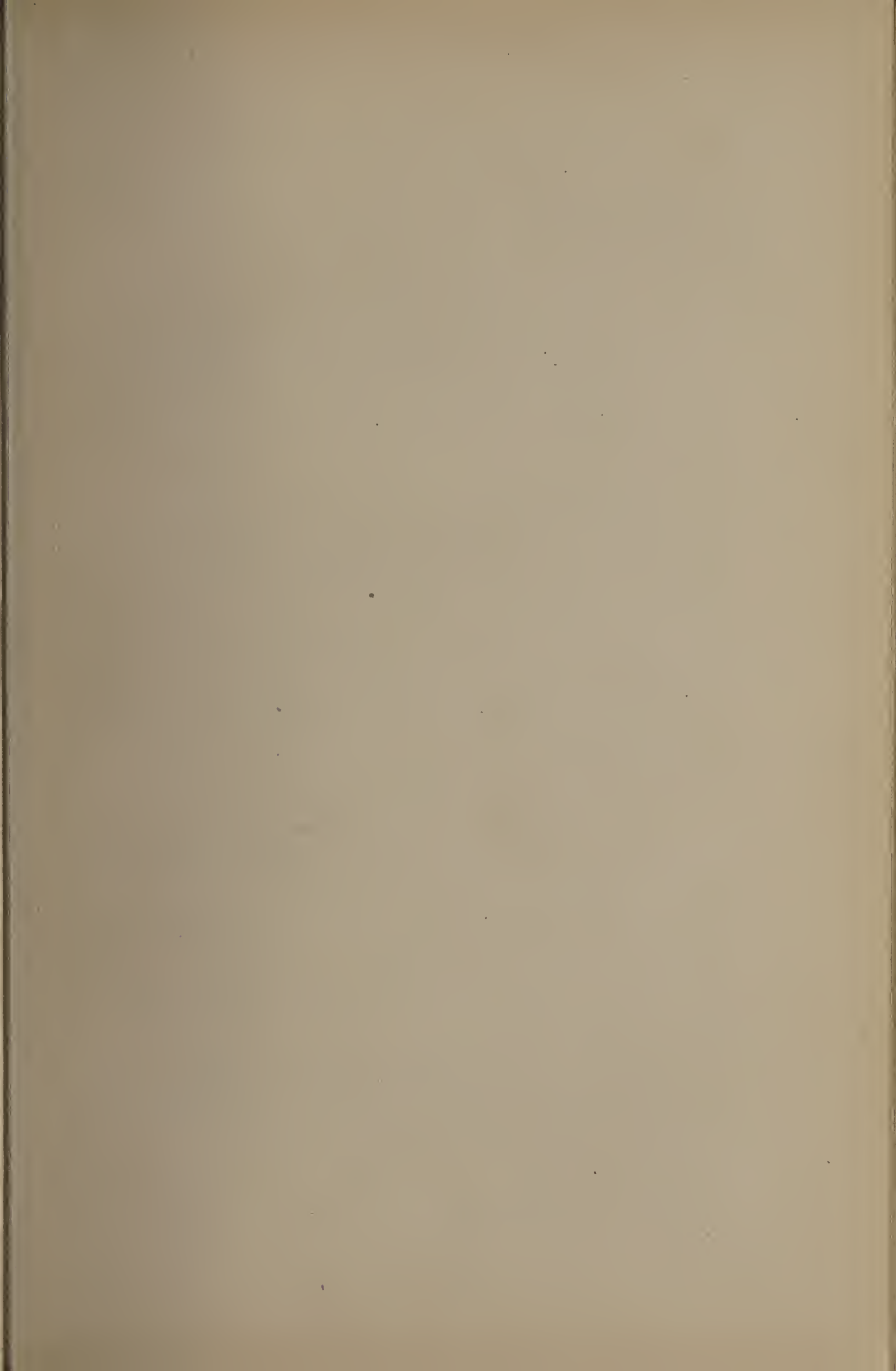
47. Use NaOH solution.

48. Use NH_4OH solution.

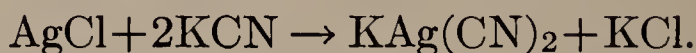
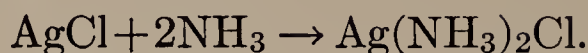
Note. Silver forms the complex ion $\text{Ag}(\text{NH}_3)_2$ with ammonia:



49. Use dilute HCl . Divide the precipitate into three portions. Expose one to the light, add NH_4OH to



the second, and KCN to the third. Acidify the *ammonia* solution with HNO_3 :



Note. Argentous chlorid, Ag_2Cl , forms by the action of light.

50. Precipitate some AgCl and then add $\text{Na}_2\text{S}_2\text{O}_3$ solution:



51. Use K_2CrO_4 solution. Test the solubility of the precipitate in $\text{HC}_2\text{H}_3\text{O}_2$ and in HNO_3 .

52. Use Na_2HPO_4 solution.

53. Use H_2S solution. Test the solubility of the precipitate in dilute and concentrated HNO_3 .

54. To three test-tubes containing AgNO_3 solution add in turn KCl , KBr , and KI . Add NH_4OH to each. Compare results.

LEAD

55. Mix equal bulks of litharge and dry sodium carbonate and heat in the reducing flame on charcoal. Show your result.

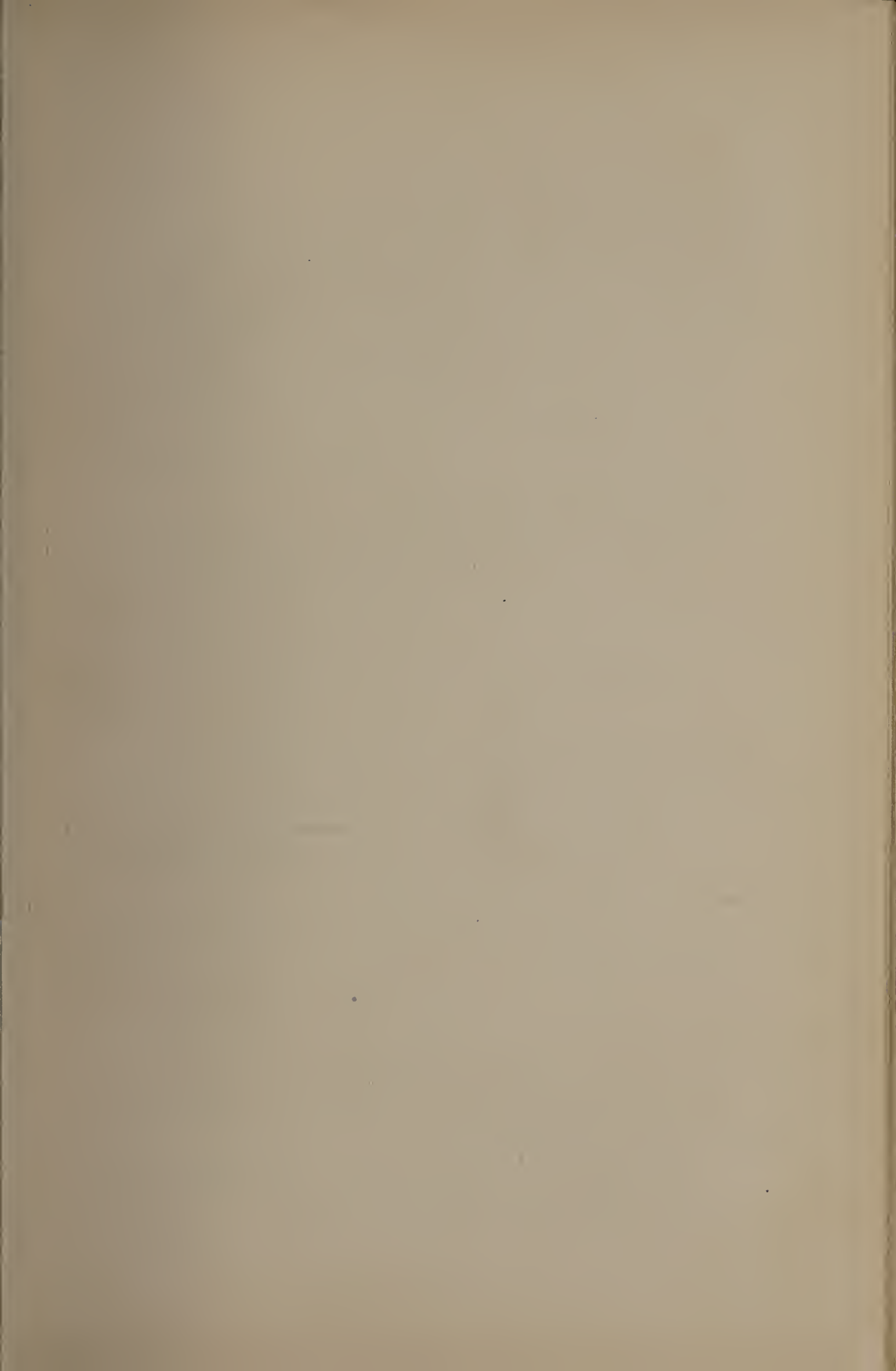
56. Test the solubility of metallic lead in dilute HCl , dilute H_2SO_4 , and dilute HNO_3 .

57. Heat a piece of metallic lead in the oxidizing flame on charcoal.

In each of the following experiments use 5 cc. of $\text{Pb}(\text{NO}_3)_2$ solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

58. Use NH_4OH .

Note. $\text{Pb}(\text{OH})_2 \cdot \text{PbO}$ forms.



59. Use dilute HCl. Filter, wash, and save the filtrate for No. 60. Divide the precipitate into two parts. Add NH_4OH to the first part on a filter paper, and with the second part repeat Experiment 33. To the ammonia filtrate add HNO_3 to acid reaction. (Compare this result with that in Experiment 49.)

60. To the filtrate from Experiment 59 add H_2S .

61. Dissolve 5 grams of lead acetate in a 500 cc. beaker *full* of water. Take 10 cc. of this solution and dilute it with 500 cc. of water. Take 100 cc. of this latter solution and pass H_2S through it.

62. Precipitate some PbS , filter, and test the solubility of the sulfid in dilute and strong (boiling) HNO_3 :



63. Use dilute H_2SO_4 . Use Na_2SO_4 solution.

64. Use KI solution. After adding the precipitant, add an equal bulk of water, boil, filter if necessary, and allow to cool. (Compare with Experiment No. 33.)

65. Use K_2CrO_4 . Note the color of the precipitate, then add a little NaOH and warm.

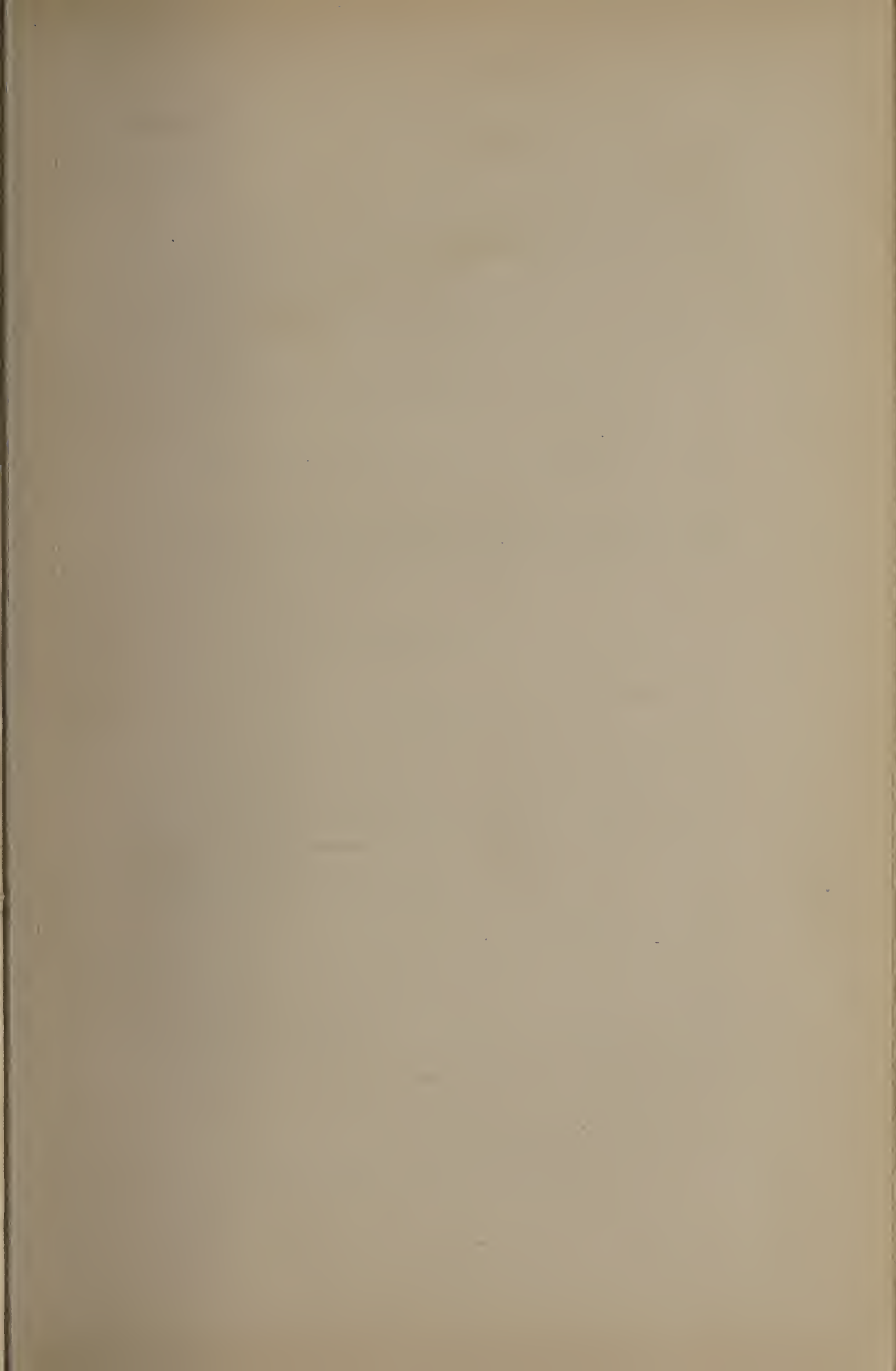
Note. By the latter reaction "chrome red," basic lead chromate ($\text{PbCrO}_4 \cdot \text{PbO}$), forms.

MERCURY

General Experiments

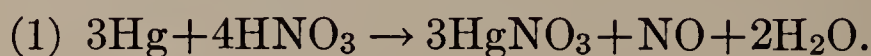
66. Test the solubility of metallic mercury in dilute HCl , H_2SO_4 , and HNO_3 .

67. Heat HgO in a dry test-tube. Heat HgCl_2 with an equal bulk of soda-lime in a dry tube.



68. Place a strip of clean copper in a solution of $\text{Hg}(\text{NO}_3)_2$ acidified with a few drops of HNO_3 .

69. In separate test-tubes dissolve mercury in HNO_3 (1 : 1). In the first have the metal in excess, in the second have the acid in excess. To each of the tubes add NaOH to alkaline reaction:



70. Place 5 cc. each of HgNO_3 and $\text{Hg}(\text{NO}_3)_2$ in separate test-tubes and add HCl to each.

MERCURY

Valence = 1

71. Test the solubility of calomel in hot and cold water.

In each of the following experiments use 5 cc. of HgNO_3 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

72. Use dilute HCl . Filter, wash, and add NH_4OH to the precipitate.

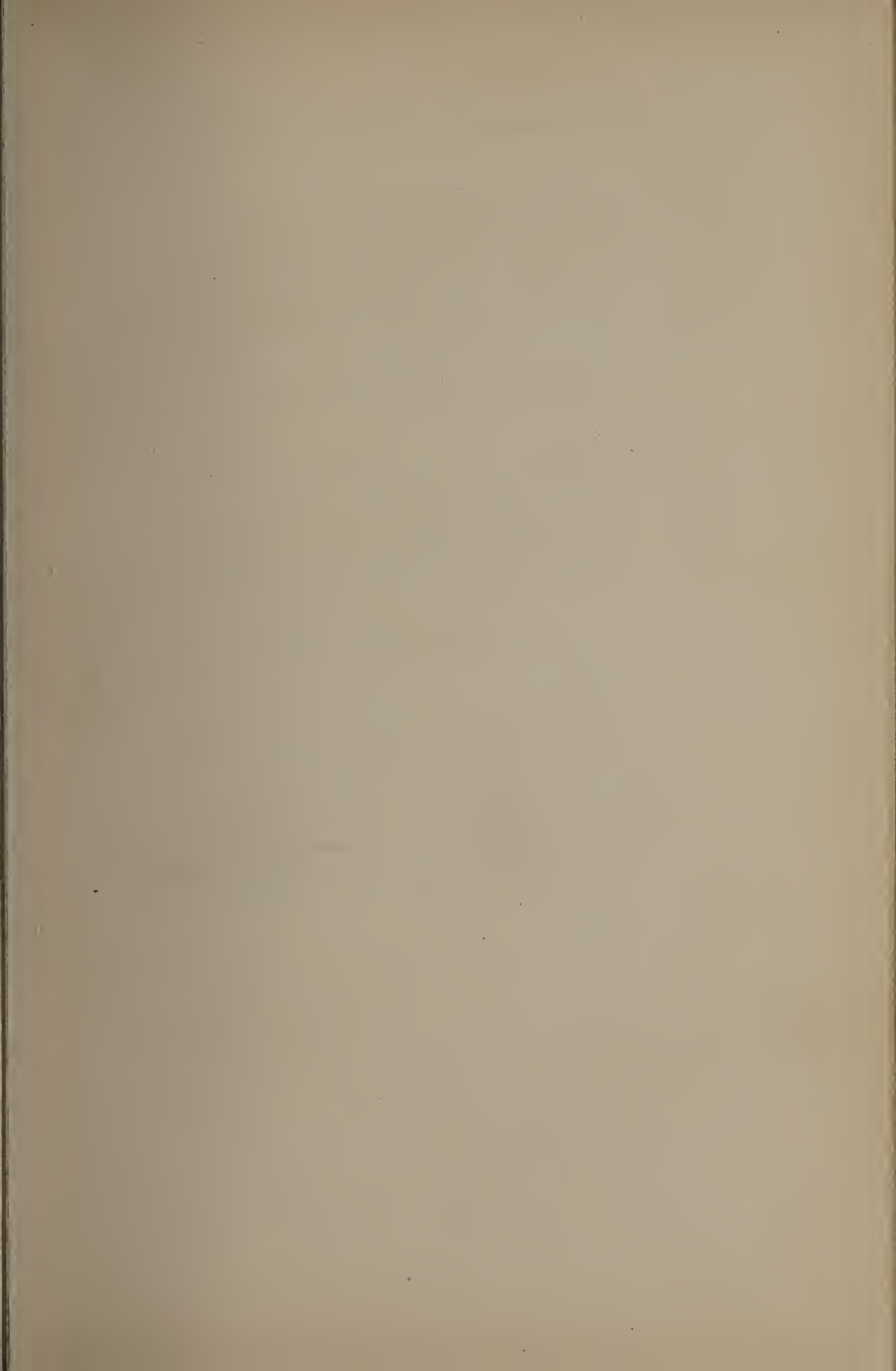


To the ammonia filtrate add HNO_3 to acid reaction. (Compare this result with that in Experiment 49.)

73. Use H_2S .

Note. Mercuric sulfid forms:





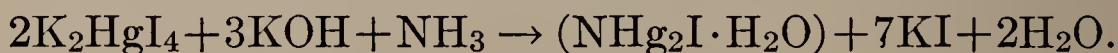
MERCURY

Valence = 2

74. To a solution of albumin add a few drops of corrosive sublimate.

75. Test the solubility of mercuric chlorid in hot and cold water.

76. Add 2 drops of NH_4OH to 500 cc. of water. To a test-tube of this solution add a few drops of Nessler's reagent: (This is a solution of HgI_2 in KI and KOH).



77. Heat 0.5 gram $\text{Hg}(\text{NO}_3)_2$ in a closed tube:



78. Add dilute HCl to 5 cc. of a solution of $\text{Hg}(\text{NO}_3)_2$ diluted with 10 cc. of water.

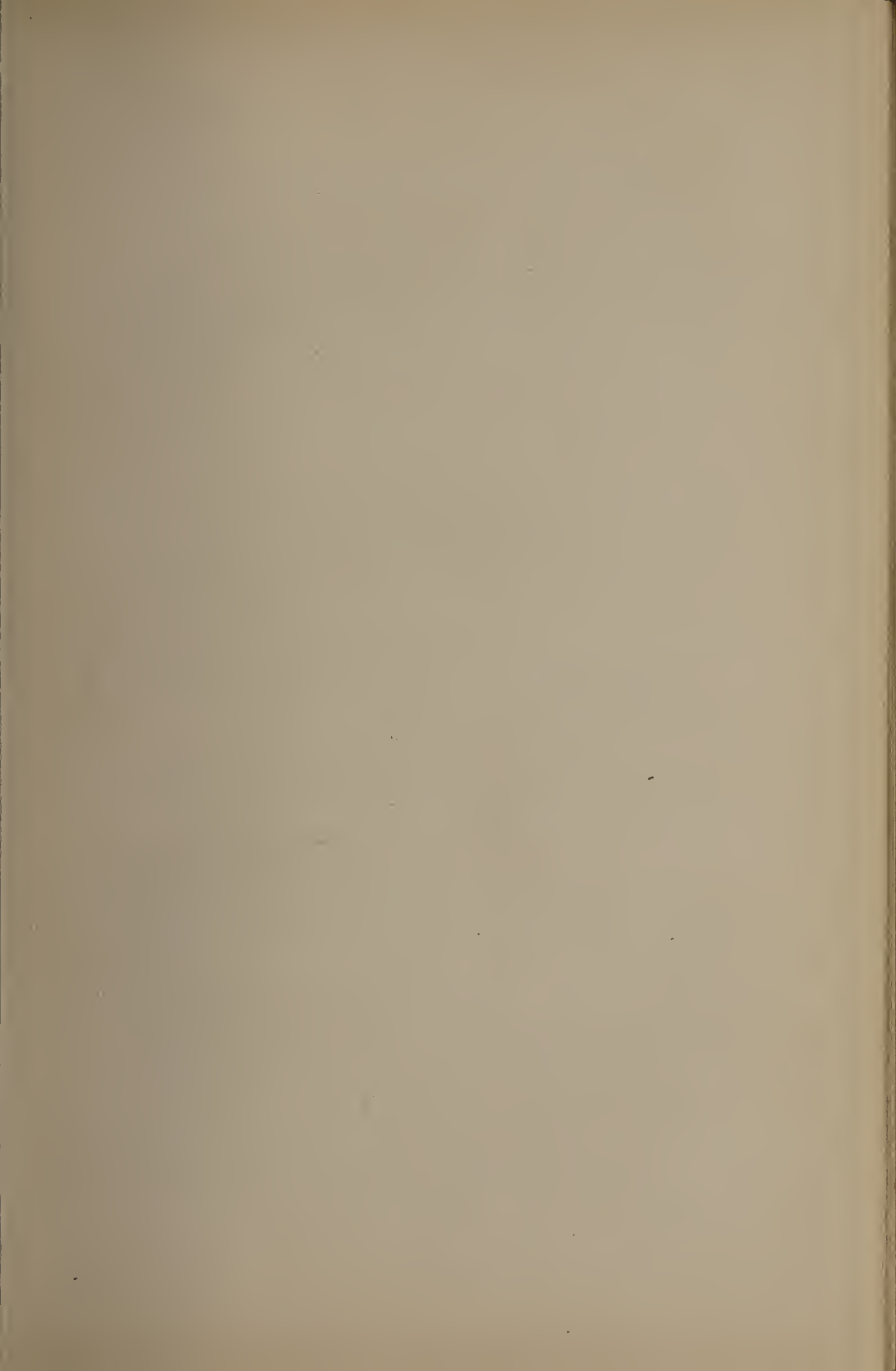
In each of the following experiments use 5 cc. of HgCl_2 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

79. Use NH_4OH :



80. Precipitate HgS and test the solubility of the sulfid in dilute $(\text{NH}_4)_2\text{S}_x$, dilute HCl , dilute HNO_3 , and *aqua regia*. Notice any change of color during the precipitation:





81. Use KI solution. Recall Experiment 76.

Note. With an excess of KI there is formed $2\text{KI} \cdot \text{HgI}_2$.

82. Use SnCl_2 solution. Add this reagent to two portions of HgCl_2 ; in the second case use but 1 or 2 cc. of HgCl_2 and a large excess of SnCl_2 . Compare your results.

BISMUTH

83. Test the solubility of metallic bismuth in dilute nitric acid, dilute hydrochloric acid, and dilute sulfuric acid.

84. Heat 0.5 gram of BiOCl with dry Na_2CO_3 on charcoal. Use the reducing flame.

85. Add a few drops of BiCl_3 to a test-tube of water. Test the solubility of the new compound in tartaric acid. (Compare with Experiment 133.)

Note. The trivalent bismuth hydroxid is very weakly basic, therefore its salts are hydrolytically decomposed by water with the formation of almost insoluble basic salts.

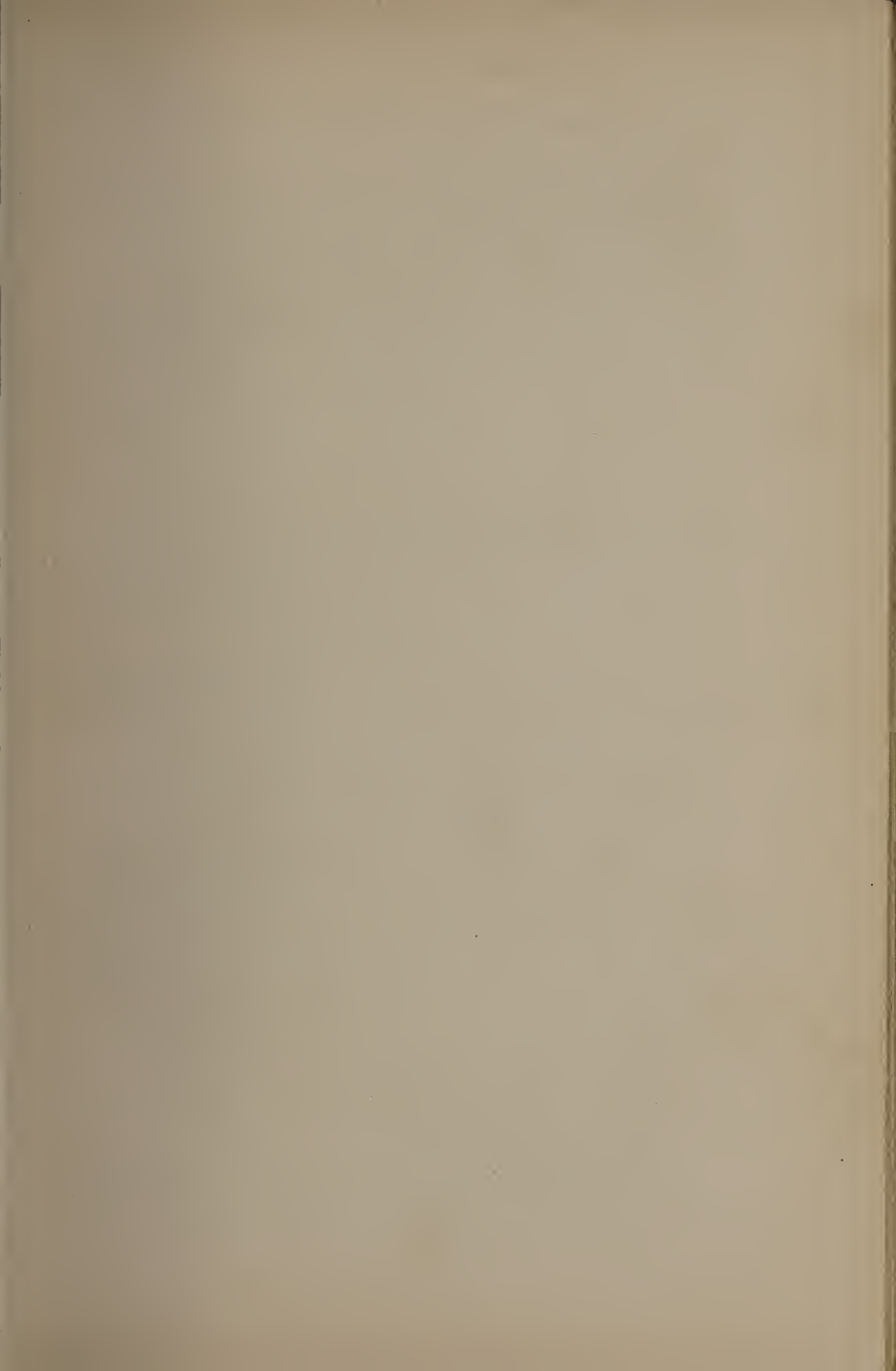
In each of the following experiments use 5 cc. of $\text{Bi}(\text{NO}_3)_3$ solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

86. Use H_2S . Test the solubility of the precipitate in hot and cold dilute HNO_3 and in dilute $(\text{NH}_4)_2\text{S}_x$.

87. Use NaOH .

88. Use NH_4OH . (Compare with Experiments 101 and 111.)

89. Add an acid solution of quinine sulfate to an acidified solution of $\text{Bi}(\text{NO}_3)_3$, then add a few drops of KI solution.



90. Prepare potassium stannite (K_2SnO_2) by adding enough KOH to 5 cc. of SnCl_2 to re-dissolve the precipitate formed. Add some of this solution to 5 cc. of $\text{Bi}(\text{NO}_3)_3$ solution.

Note. This reaction is very delicate, is one of reduction, and forms bismuthous oxid, BiO .

COPPER

General Experiments

91. Test the solubility of metallic copper in hot and cold, dilute and concentrated, nitric, hydrochloric, and sulfuric acids.

92. Mix equal weights of CuCl_2 and dry Na_2CO_3 , and heat in the reducing flame on charcoal.

93. Heat strongly a piece of metallic copper in the blast-lamp. While it is still hot, immerse it in cold water. Observe whether more than one product has resulted.

94. Prepare a borax bead, place upon it a little CuCl_2 , and heat again in the oxidizing flame. Observe its color when hot and cold.

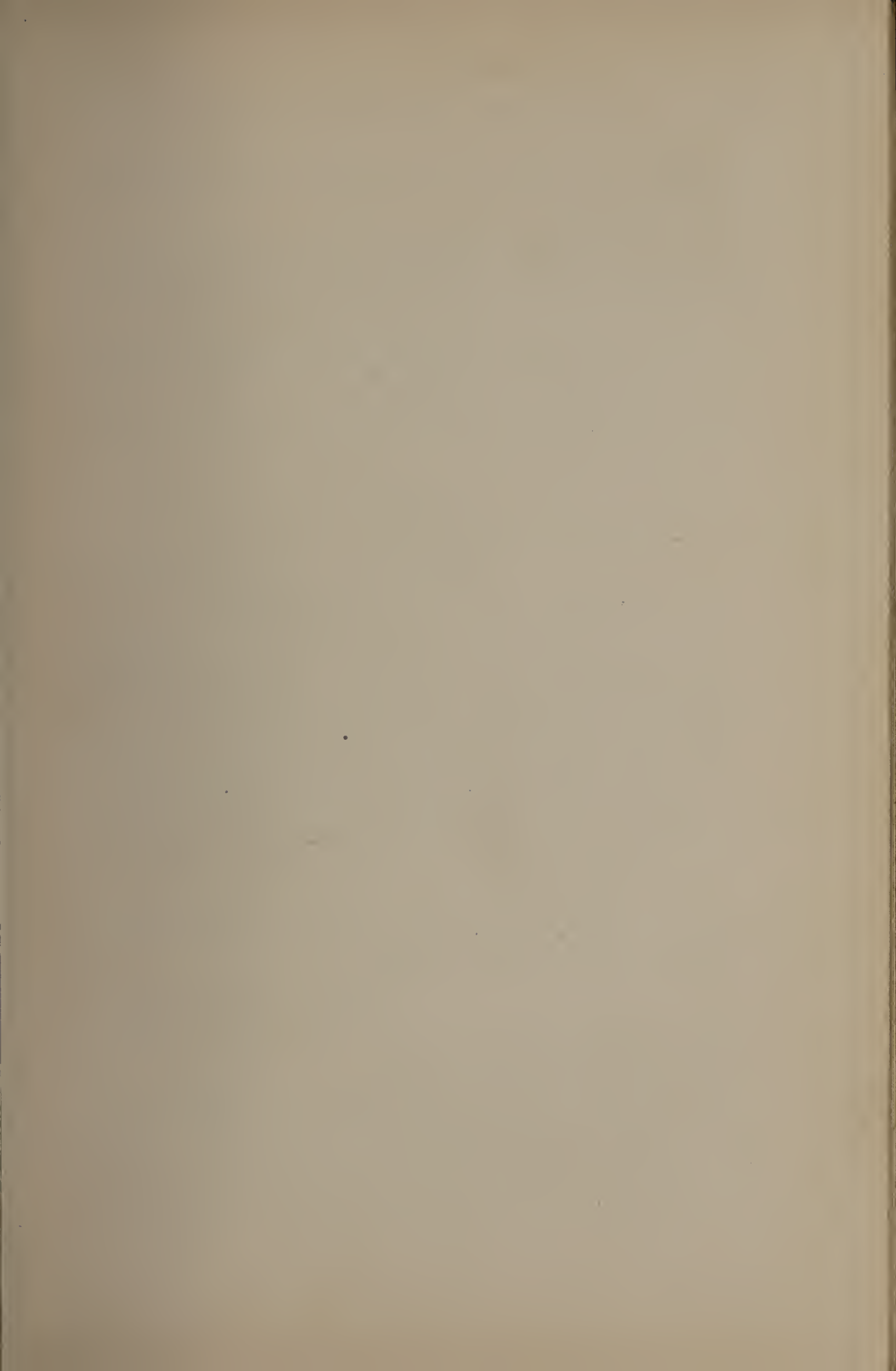
95. (a) Make a flame test with $\text{Cu}(\text{NO}_3)_2$ on a platinum wire. (b) Dip the wire into concentrated HCl on a watch-glass before touching it to the copper salt.

96. Dissolve 1 gram of metallic copper in the smallest possible amount of 1 : 1 HNO_3 . Evaporate the solution to dryness and ignite.

COPPER

Valence = 1

97. To 10 cc. of CuSO_4 solution add 3 grams of tartaric acid. Stir until it is dissolved and then add an excess



of KOH. Add 1 gram of either glucose or milk-sugar and boil the mixture.

Note. The yellowish precipitate at first formed is hydrated cuprous oxid ($4\text{CuO} \cdot \text{H}_2\text{O}$) and this, by further heating, is converted to red cuprous oxid.

98. Add a considerable bulk of cold water to a solution of CuCl in concentrated HCl .

99. Add KOH to a solution of CuCl . Observe the color of the precipitate and then boil.

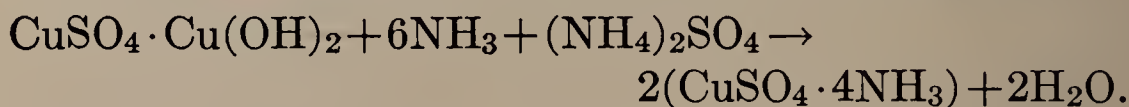
COPPER

Valence = 2

100. Immerse a strip of zinc in CuSO_4 solution.

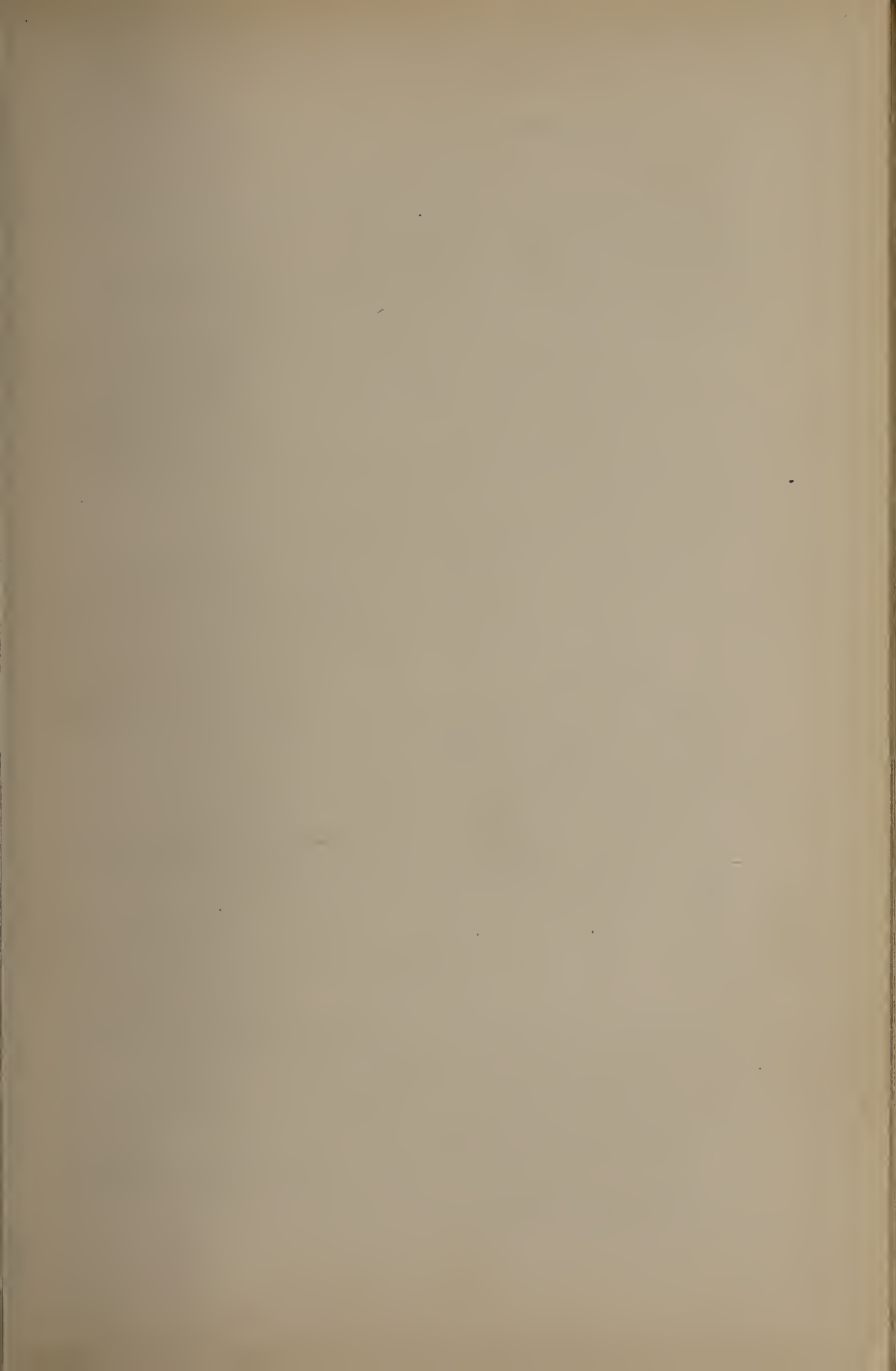
In each of the following experiments use 5 cc. of CuSO_4 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

101. Use *very dilute* NH_4OH (compare with Experiment 88). After noting the effect of an excess of the reagent, add ethanol until a precipitate results. (Recall Experiments 21 and 22.) Filter this precipitate and examine it. Try its solubility in water.



102. (a) Use NaOH . (b) Have the CuSO_4 solution boiling before adding the NaOH .

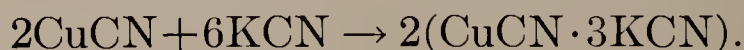
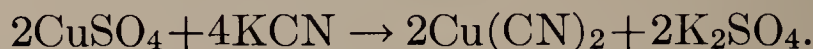
103. Use H_2S . Filter, and examine the solubility of the precipitate in dilute HCl and hot dilute HNO_3 . Also try the effect of dilute $(\text{NH}_4)_2\text{S}_x$ and dilute K_2S_x . In the case of the two latter reagents, filter after treatment,



and neutralize the filtrates with HCl. Compare the results with that obtained when 1 cc. of $(\text{NH}_4)_2\text{S}_x$ in 10 cc. of water is acidified with HCl.

104. Use KCN. After an excess of KCN has been added, add H_2S .

Note. The compound formed by the excess of the reagent is colorless potassium cuprous cyanide, $\text{CuCN} \cdot 3\text{KCN}$. This compound in solution is dissociated into the ions 3K^+ and $\text{Cu}(\text{CN})_4^{3-}$, and, owing to the fact that the complex anion $\text{Cu}(\text{CN})_4^{3-}$ is practically not dissociated, no precipitate results with H_2S . (Compare with Experiment 113).



105. Add 3 or 4 drops of CuSO_4 solution to a test-tube of water. Acidify the solution with acetic acid and then add a few drops of $\text{K}_4\text{FeC}_6\text{N}_6$.

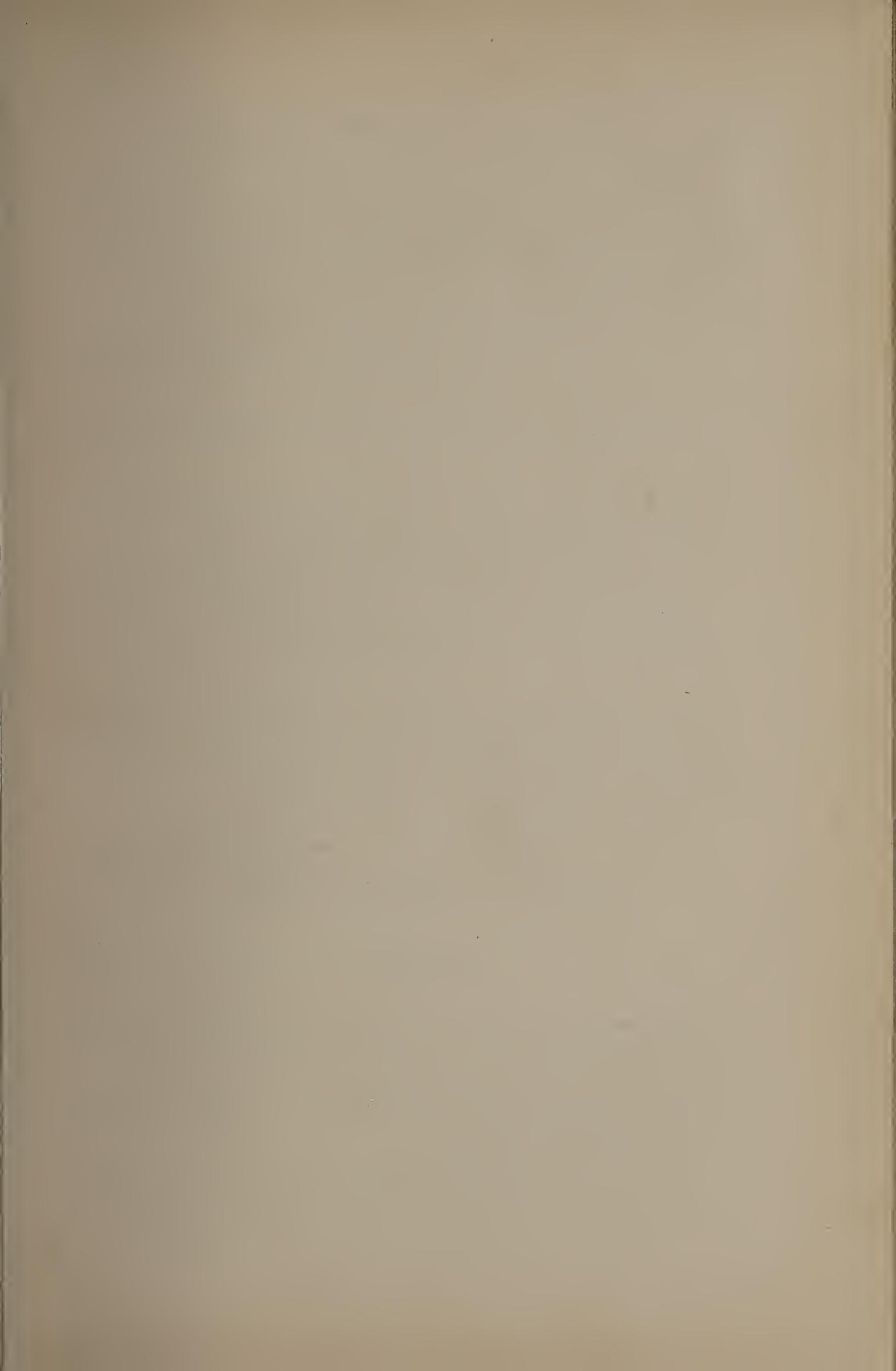
106. Leave some moist CuS on a filter-paper until the next laboratory period. Then put the paper and precipitate into a beaker containing 50 cc. of water. Warm the water, filter, and to the filtrate add NH_4OH .

CADMIUM

107. Test the solubility of metallic cadmium in dilute HCl, dilute H_2SO_4 , and dilute HNO_3 .

108. Mix 0.5 gram of CdCl_2 with dry Na_2CO_3 and heat on charcoal in the reducing flame.

109. Introduce a strip of zinc into 10 cc. of a solution of CdCl_2 to which two or three drops (*only*) of dilute HCl have been added.



In each of the following experiments use 5 cc. of a solution of CdSO_4 or $\text{Cd}(\text{NO}_3)_2$, diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

110. Use NaOH .

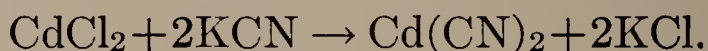
111. Use *very dilute* NH_4OH . (Compare with Experiment 88.)

Note. The compound formed by an excess of the reagent, supposing CdSO_4 was used, is $\text{Cd}(\text{NH}_3)_4\text{SO}_4$.

112. Use H_2S . Before passing in the gas, acidify the cadmium solution with a *few drops* of HCl . Filter, and test the solubility of the precipitate in dilute HNO_3 , dilute HCl , dilute H_2SO_4 , and dilute $(\text{NH}_4)_2\text{S}_x$.

113. Use KCN . After an excess of KCN has been added, add H_2S . (Compare with Experiment 104.)

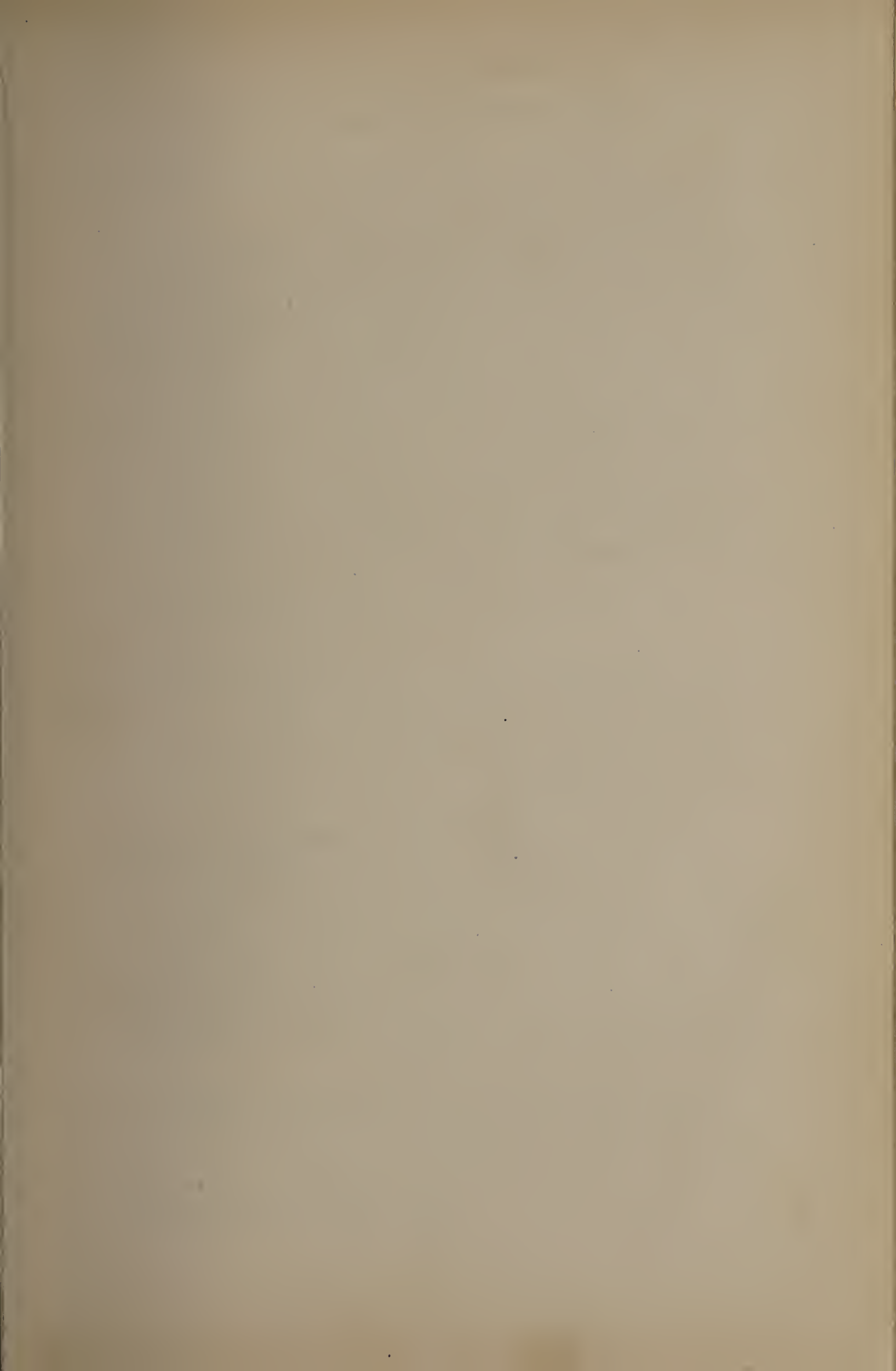
Note. The compound formed by the excess of the reagent is $2\text{KCN} \cdot \text{Cd}(\text{CN})_2$. Since the complex anion $\text{Cd}(\text{CN})_4^{=}$ is itself more or less dissociated into Cd^{++} and 4CN^- , sulfuretted hydrogen precipitates CdS from a solution of this compound.



ARSENIC

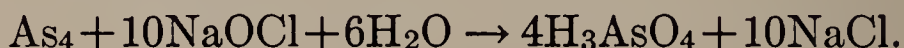
114. Place a minute quantity as As_2O_3 in the closed, drawn-out point of a piece of glass tubing. Just above the arsenic oxid place a fragment of charcoal. Heat the tube where the charcoal is and then immediately heat the point of the tube.

115. Place a very small amount of As_2O_3 in a glass tube 10 cm. long and open at both ends. Heat the powder



while the tube is held in a sloping position. Determine the crystalline structure of the deposit.

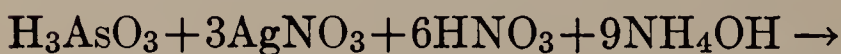
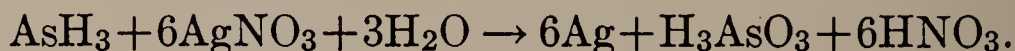
116. [*Under the hood.*] Arrange a hydrogen generator, dryer, and a constricted tube as shown in the model on the blackboard. Use C. P. zinc and dilute H_2SO_4 . When the gas is pure, light it at the jet. Now add a *few drops* of the special dilute solution of As_2O_3 in HCl . Notice any change in the color of the flame and then hold a porcelain dish in the jet of burning gas. Make several "spots." Try the effect of heat on one, and add to the others, separately, NaOCl , $(\text{NH}_4)_2\text{S}$ (1 drop only), and hot HNO_3 . Also heat the delivery tube just behind one of the constrictions:

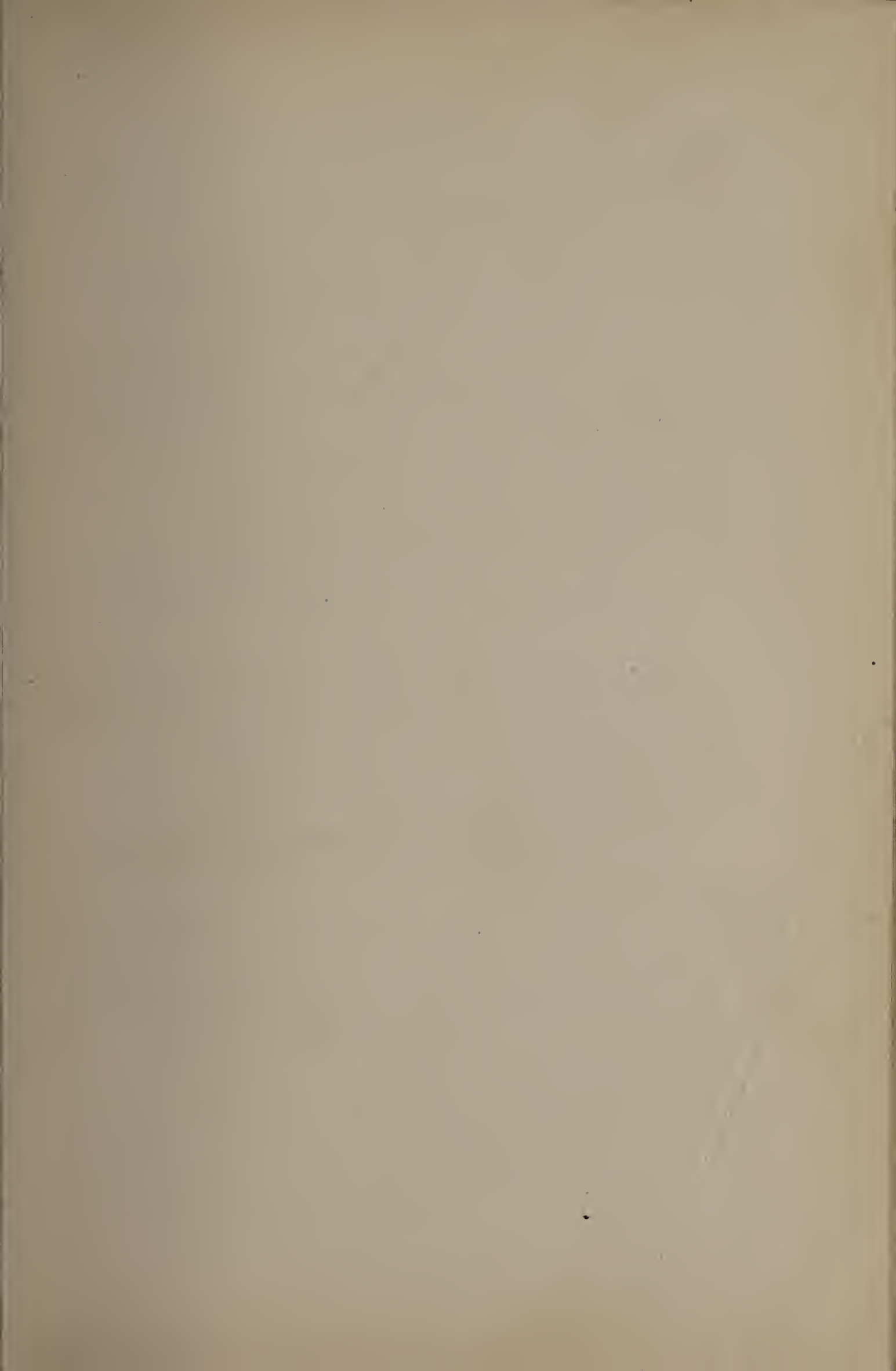


Record the properties of the spots and mirrors as indicated in Experiment 131. [This is Marsh's test.]

Note. After the flow of gas has ceased, wash out the generator in the sink under the hood.

117. Use the same precautions as above and add even less of the arsenic solution. Have the hood window nearly closed. Use the same generator but omit the dryer and the constricted tube. Pass the stream of AsH_3 into a solution of AgNO_3 . When the flow of gas stops, filter the solution, add to it a little more AgNO_3 , and carefully neutralize it with dilute NH_4OH . Observe any formation at the junction of the two liquids:

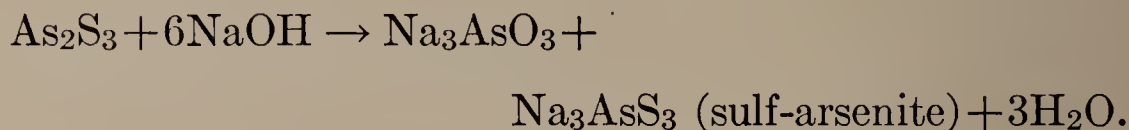




118. Slightly acidify 10 cc. of a solution of Na_3AsO_3 , place in it a piece of clean sheet copper, and warm.

Note. This is *Reinsch's test*, available for arsenious compounds but not for arsenic compounds unless they are first boiled with strong HCl or with H_2SO_3 . As_2Cu_5 forms.

119. Into each of two test-tubes place 5 cc. of a solution of Na_3AsO_3 . Make one acid with HCl , and the other alkaline with NaOH . Pass H_2S into both tubes. Now acidify with HCl the one that is alkaline:



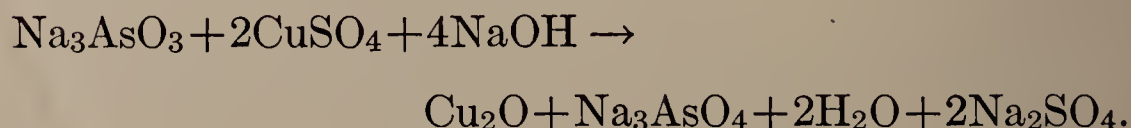
120. Pass H_2S into 5 cc. each of solutions of Na_3AsO_3 , and Na_3AsO_4 , both of which have been acidified with HCl . (See Experiment 125.) Repeat this experiment without the addition of HCl .

121. Add AgNO_3 solution to 5 cc. each of solutions of Na_3AsO_3 and Na_3AsO_4 .

122. To 5 cc. of a solution of Na_3AsO_3 add CuSO_4 solution:



123. Add a large excess of NaOH to 10 cc. of a solution of Na_3AsO_3 , add a few drops of CuSO_4 , and warm:



(In your notes, state in what way the arsenite has acted here.)

124. Make 5 cc. of a solution of Na_3AsO_4 alkaline with NH_4OH and then add "magnesia mixture." Allow the mixture to stand if necessary.

Note. NH_4OH in excess insures the insolubility of $\text{NH}_4\text{MgAsO}_4$, and NH_4Cl prevents the precipitation of $\text{Mg}(\text{OH})_2$:

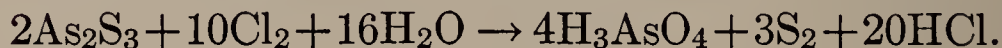
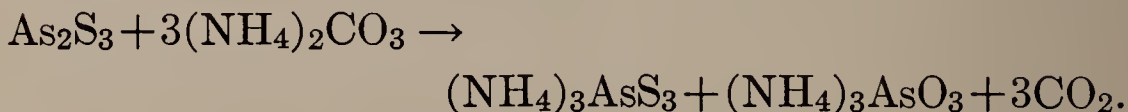
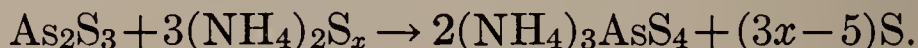


125. Acidify 10 cc. of a solution of Na_3AsO_4 with HCl , dilute, and bring the mixture to a boil. With the solution still boiling, pass in H_2S gas until a precipitate is obtained:

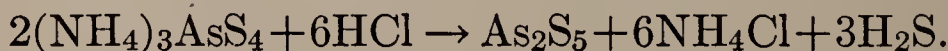


Note. Explain why a precipitate does not form immediately. Remember that the precipitate is As_2S_3 and not As_2S_5 as might be expected.

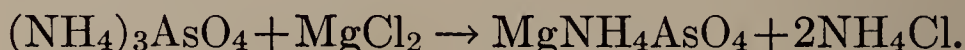
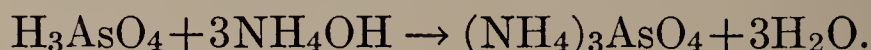
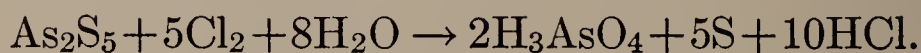
126. Precipitate As_2S_3 from 15 cc. of a solution of As_2O_3 in HCl . Filter, and test the solubility of the precipitate in hot, strong HCl , $(\text{NH}_4)_2\text{S}_x$ (compare with Experiments 80 and 103), $(\text{NH}_4)_2\text{CO}_3$, and boiling, strong $\text{HCl} + \text{KClO}_3$:



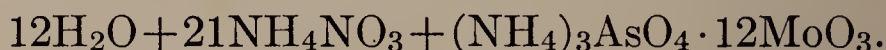
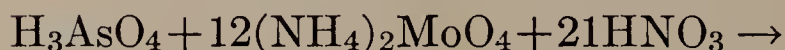
127. Place 5 cc. of $(\text{NH}_4)_2\text{S}_x$ in each of two test-tubes and dilute each with 10 cc. of water. In one tube dissolve some As_2S_3 and then acidify both solutions with HCl :



128. Prepare some As_2S_5 as in Experiment 127. Dissolve this in hot, concentrated HCl and KClO_3 . Make this solution strongly alkaline with NH_4OH and then add magnesia mixture: (Compare with Experiment 124.)



129. Acidify 10 cc. of a solution of Na_3AsO_4 with HNO_3 and then add $(\text{NH}_4)_2\text{MoO}_4$ solution. Allow the mixture to stand for some time, and then heat:



Compare this result with that obtained by starting with Na_2HPO_4 solution.

ANTIMONY

130. Try the solubility of metallic antimony in concentrated and dilute HCl , H_2SO_4 , and HNO_3 . Also try *aqua regia*:

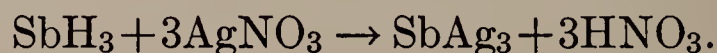


131. Repeat Marsh's test as in Experiment 116. Use fresh CaCl_2 in the dryer and be sure that all parts of the apparatus have been thoroughly freed from arsenic. This time put a piece of platinum foil on the top of the zinc in the generator. Use a dilute solution of SbCl_3 .

Compare the spots and mirrors of As and Sb as follows:

	Arsenic.	Antimony.
Lustre.....		
Volatility.....		
Solubility.....		
Color formed by $(\text{NH}_4)_2\text{S}$		

132. Repeat Experiment 117, using SbCl_3 solution instead of a solution of As_2O_3 in HCl . Filter as before, but neglect the filtrate and treat the precipitate as follows: Thoroughly wash the substance on the filter-paper, and then dissolve it by boiling in a strong solution of tartaric acid to which a few drops of HNO_3 have been added. Filter, acidify with HCl , and pass H_2S through the warmed solution:



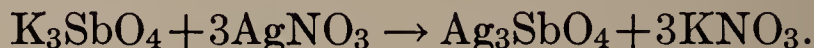
133. Add 2 cc. of SbCl_3 to a test-tube full of water. Try the solubility of the new compound in tartaric acid. (Compare with Experiment 85.)

134. To 5 cc. of SbCl_3 add NaOH , at first in small quantity, and then in excess:



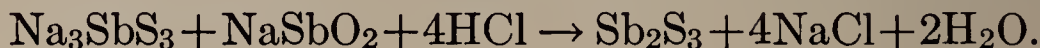
135. Add AgNO_3 solution to 5 cc. each of solutions of

K_3SbO_3 and K_3SbO_4 . Try the solubility of both precipitates in NH_4OH :

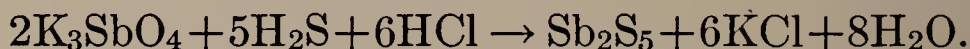


Note. The precipitate from K_3SbO_3 consists of Ag_2O and metallic silver.

136. Place 5 cc. of a solution of tartar emetic [$KSbO(C_4H_4O_6)$] in each of two test-tubes. Make one slightly acid with HCl and the other strongly alkaline with $NaOH$. Saturate both with H_2S . Now acidify with HCl the one which was alkaline:

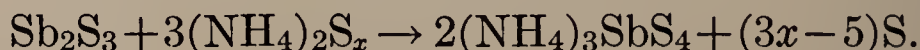


137. Acidify 10 cc. of K_3SbO_4 solution with HCl and pass into it H_2S gas: (Compare with Experiment 125).



Note. The above reaction is true in part, but the precipitate also contains some Sb_2S_3 and free sulfur.

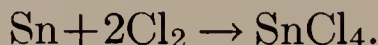
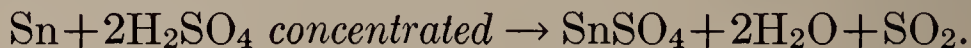
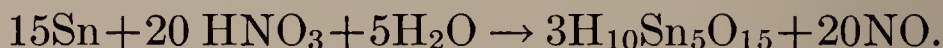
138. Precipitate Sb_2S_3 from 15 cc. of $SbCl_3$. Filter, and test the solubility of the precipitate in hot, strong HCl , also in $(NH_4)_2S_x$ and $(NH_4)_2CO_3$: (Compare with Experiment 126).



139. Repeat Experiment 127, using Sb_2S_3 instead of As_2S_3 .

TIN

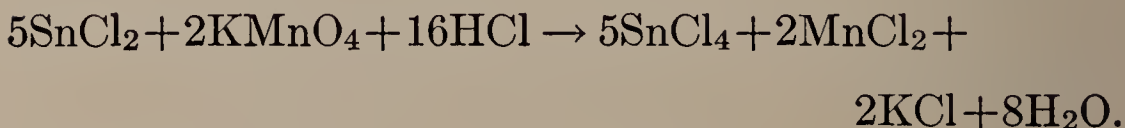
140. Test the solubility of metallic tin in dilute and concentrated, hot and cold, nitric, hydrochloric, and sulfuric acids. Also try *aqua regia*:



141. Dilute 20 cc. of SnCl_2 with an equal volume of water. Acidify the solution with HCl , and place in it a strip of metallic zinc which is in contact with a piece of platinum foil. Remove the deposit which forms on the zinc, dissolve it in strong HCl , and to this solution add HgCl_2 . Notice whether there is any deposit on the platinum.

142. In each of two test-tubes place 5 cc. each of SnCl_2 and SnCl_4 . Pass H_2S through both solutions.

143. Dilute 1 cc. of SnCl_2 with 10 cc. of water, add 5 cc. of chlorin water and boil. Now pass H_2S into the solution. Repeat, using KMnO_4 instead of chlorin water. Add the KMnO_4 until a faint color is obtained:



144. Dilute 2 cc. of SnCl_2 with 10 cc. of water and add a few drops of HgCl_2 . Now add to a few drops of SnCl_2 an

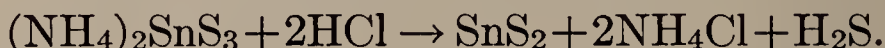
excess of HgCl_2 . Write *both* reactions. Repeat, using SnCl_4 .

145. Strongly acidify with HCl 1 cc. of SnCl_4 solution diluted with 10 cc. of water. Add some copper turnings, boil for several minutes, and then decant the clear liquid. Now add a few drops of HgCl_2 .

146. Precipitate SnS from 10 cc. of SnCl_2 solution. Filter, wash, and test the solubility of the sulfid in hot, concentrated HCl and dilute $(\text{NH}_4)_2\text{S}_x$.

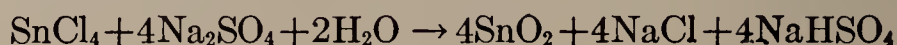
147. Repeat, starting with SnCl_4 .

148. Repeat Experiment 139, using SnS instead of Sb_2S_3 :



149. Dilute 10 cc. of a neutral solution of SnCl_4 with 10 cc. of water, add 10 cc. of Na_2SO_4 solution, and boil.

Note. Stannic sulfate first forms and is then hydrolyzed by the action of the water:

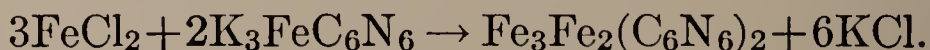


or

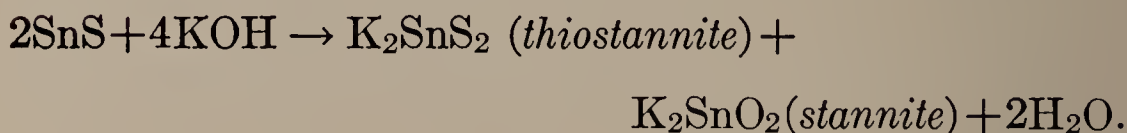
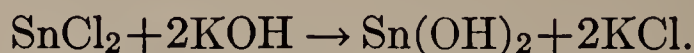


150. Dilute 5 cc. of SnCl_2 solution with 10 cc. of water. Add to this a few drops of sodium nitroprusside solution. Now add a *few drops* of HCl , avoiding a large excess of acid.

151. Add to 2 cc. of FeCl_3 , diluted with 10 cc. of water, a few drops of $\text{K}_3\text{FeC}_6\text{N}_6$. Note whether there is any precipitate and then add SnCl_2 :



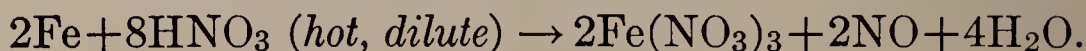
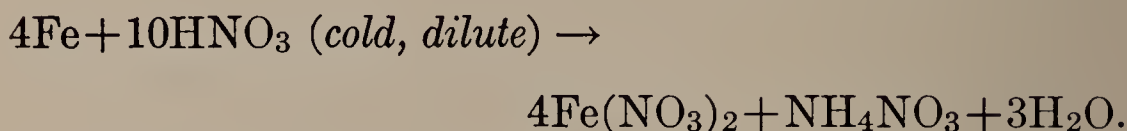
152. Pass H_2S into 5 cc. of SnCl_2 made alkaline with an *excess* of KOH . Then acidify the solution with HCl :



IRON

General Experiments

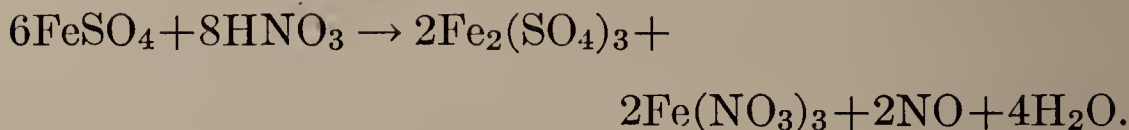
153. Try the solubility of metallic iron in hot and cold, dilute hydrochloric, sulfuric, and nitric acids:



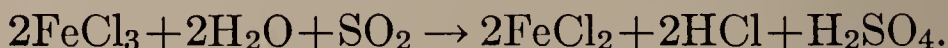
154. Prepare a borax bead and heat it with FeSO_4 in the reducing flame. Repeat, using the oxidizing flame.

155. Cover 5 grams of iron with 50 cc. of dilute H_2SO_4 . Dilute the liquid with 50 cc. more of water and warm gently until the evolution of gas slackens, keeping the volume up to the original amount. Filter, and divide the filtrate into two portions. Keep one portion for work with *ferrous* ion and divide the other into three portions. Add to these three portions, separately: (a) a few drops of HNO_3 ; (b) 10 cc. of bromin water; (c) 10 cc. of chlorin water. Notice any changes of color while they boil for a few min-

utes. Now to 5 cc. each of the ferrous solution and of (a) (b), and (c) diluted with 10 cc. of water, add a few drops of KSCN solution:



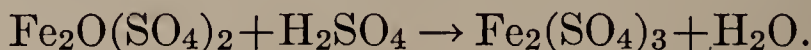
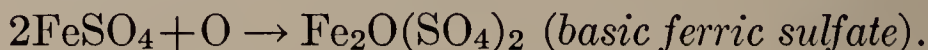
156. Dilute five portions of 2 cc. each of FeCl_3 with 10 cc. of water. To four of these solutions add, separately, (a) HCl and a few fragments of zinc, (b) SnCl_2 , (c) H_2SO_3 , (d) H_2S . Warm the first three for some minutes and then add $\text{K}_3\text{FeC}_6\text{N}_6$ to all five solutions:



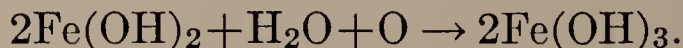
IRON

Valence = 2

157. Dissolve 3 grams of crystallized ferrous sulfate in 25 cc. of water. Filter the solution, and then allow it to stand in an evaporating dish until the next laboratory period, at which time, acidify the solution with H_2SO_4 and warm:



158. Take 20 cc. of side-shelf FeSO_4 solution in an evaporating dish and add NH_4OH in excess. Note the color of the precipitate and then allow to stand for half an hour, stir frequently, and observe any change in color of the precipitate:



In each of the following experiments use 5 cc. of FeSO_4 (prepared in Experiment 155) diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

159. Use NaOH . Notice all changes of color.

160. Use NH_4OH .

161. Use $\text{K}_4\text{FeC}_6\text{N}_6$. Notice all changes of color.

162. Use $\text{K}_3\text{FeC}_6\text{N}_6$. (*Turnbull's blue.*)

163. Use KCNS .

164. Use H_2S .

165. Use NH_4OH and $(\text{NH}_4)_2\text{S}$. Test the solubility of the precipitate in dilute HCl .

IRON

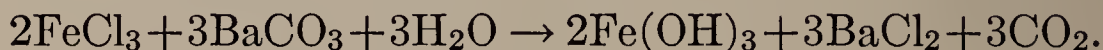
Valence = 3

In each of the following experiments use 5 cc. of FeCl_3 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

166. Use NaOH . Test the solubility of the precipitate in dilute HCl .

167. Use NH_4OH .

168. Use BaCO_3 :



169. Use $\text{K}_4\text{FeC}_6\text{N}_6$. (*Prussian blue forms.*)

170. Use $\text{K}_3\text{FeC}_6\text{N}_6$.

171. Use KCNS .

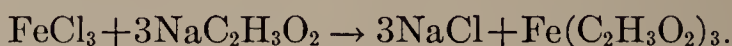
172. Use the solution made by boiling 2 grams of nutgalls in 20 cc. of water and filtering.

Note. Nutgalls contain tannic acid.

173. Dilute 10 cc. of FeCl_3 with 200 cc. of water.

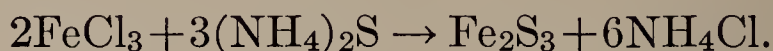
Add 10 grams of crystallized $\text{NaC}_2\text{H}_3\text{O}_2$ and boil. Notice all changes of color.

Note. $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3$ first forms and then precipitates as basic ferric acetate:



174. Pass H_2S into 10 cc. of FeCl_3 solution diluted with an equal volume of water.

175. Add $(\text{NH}_4)_2\text{S}$ to 5 cc. of FeCl_3 diluted with 10 cc. of water:



NICKEL

176. Prepare a borax bead and heat it in both the oxidizing and the reducing flame with $\text{Ni}(\text{NO}_3)_2$. Observe the color each time, both hot and cold.

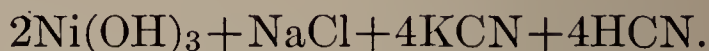
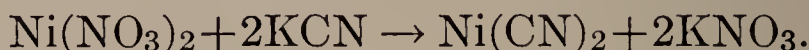
177. Precipitate NiS by adding $(\text{NH}_4)_2\text{S}$ to 10 cc. of $\text{Ni}(\text{NO}_3)_2$ solution. Filter, wash the precipitate, and test its solubility in cold, dilute HCl and *aqua regia*.

178. Repeat Experiment 177, but make the 10 cc. of the nickel salt solution strongly ammoniacal before adding the $(\text{NH}_4)_2\text{S}$. If, upon filtering, the filtrate is dark-colored, acidify it with $\text{HC}_2\text{H}_3\text{O}_2$ and warm gently.

Note. Nickel sulfid is slightly soluble in an excess of ammonia and ammonium sulfid.

179. Add 1 cc. of $\text{HC}_2\text{H}_3\text{O}_2$ to 5 cc. of $\text{Ni}(\text{NO}_3)_2$ solution. Now add an equal volume of KNO_2 solution and allow the mixture to stand several hours. (Compare with Experiment 186.)

180. To 2 cc. of $\text{Ni}(\text{NO}_3)_2$ solution add NaOH just to alkaline reaction. Now add an excess of KCN and then an equal volume of NaOCl solution (or bromin water) and warm the mixture:



Note. Observe the oxidation from a *nickelous* to a *nickelic* salt. (Compare with Experiment 187.)

In each of the following experiments, use 10 cc. of NiSO_4 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

181. Use NaOH . Afterwards add NH_4Cl solution.

182. Use *very dilute* NH_4OH . (Prepare this by adding a few drops of the regular reagent to a test-tube full of water.)

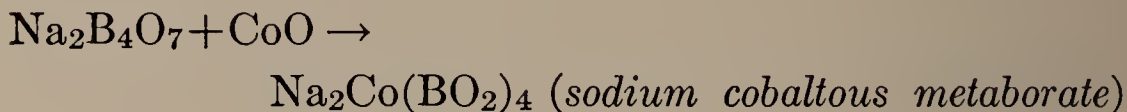
Note. Nickel forms a complex with NH_3 :



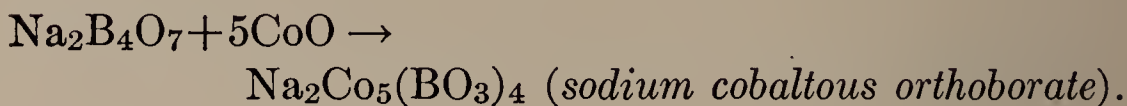
183. Repeat Experiment 182, but add 15 cc. of NH_4Cl solution before adding the NH_4OH .

COBALT

184. Prepare a borax bead and heat it in the oxidizing flame with $\text{Co}(\text{NO}_3)_2$:

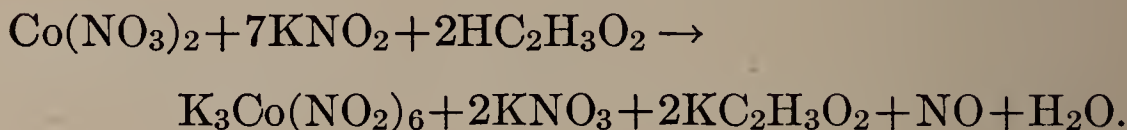


or

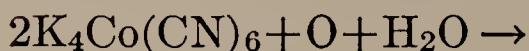
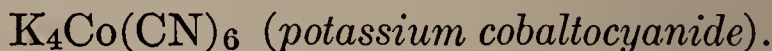
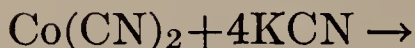
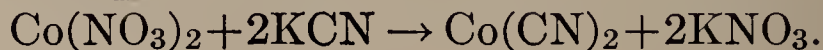


185. Precipitate CoS by adding $(\text{NH}_4)_2\text{S}$ to 10 cc. of $\text{Co}(\text{NO}_3)_2$ solution. Filter, wash the precipitate, and test its solubility in cold, dilute HCl and in *aqua regia*.

186. Add 1 cc. of $\text{HC}_2\text{H}_3\text{O}_2$ to 5 cc. of $\text{Co}(\text{NO}_3)_2$ solution. Now add an equal volume of KNO_2 solution and allow the mixture to stand several hours. (Compare with Experiment 179).



187. Repeat Experiment 180, using $\text{Co}(\text{NO}_3)_2$ solution instead of $\text{Ni}(\text{NO}_3)_2$:



188. To 5 cc. of CoCl_2 solution, acidified with HCl , add a little solid ammonium thioacetate ($\text{NH}_4\text{CH}_3\text{COS}$). Now add a few drops of SnCl_2 (*to reduce any iron present*), and then an equal volume of amyl alcohol (or of a mixture of ethanol and ethyl ether). Shake well and allow to separate.

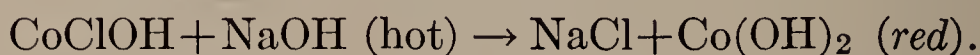
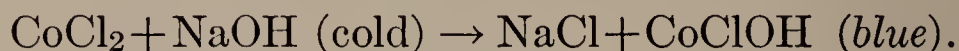
Note. The color of the upper layer is probably due to double cobalt ammonium thioacetate $(\text{CH}_3\text{COS})_2\text{CO} \cdot 2\text{CH}_3\text{COSNH}_4$. This is a most delicate test for cobalt and can be used to detect 1 part of cobalt in 500,000 parts of water.

189. Place a drop or two of CoCl_2 solution on a piece of filter-paper. Allow it to dry and then warm the paper. Now hold the paper in steam.

190. To 2 cc. of $\text{Co}(\text{NO}_3)_2$ solution add an equal volume of a dilute solution of sodium silicate.

In each of the following experiments use 10 cc. of CoCl_2 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

191. Repeat Experiment 181, using the cobalt solution in place of the nickel solution. After observing the color of the original precipitate, warm the mixture:



192. Repeat Experiment 182, using the cobalt solution.

193. Repeat Experiment 183, using the cobalt solution.

CHROMIUM

194. Prepare a borax bead and heat it in the reducing flame with a chromium salt.

Note. The compound formed is a metaborate, $\text{Na}_6\text{Cr}_2(\text{BO}_2)_{12}$.

195. Mix together thoroughly 0.5 gram of Cr_2O_3 , 1 gram of dry Na_2CO_3 , and 0.5 gram of KNO_3 . Fuse this mixture upon platinum foil. Boil the fused mass with water, filter, and divide the filtrate into two portions. Acidify one portion with $\text{HC}_2\text{H}_3\text{O}_2$ and then add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$. Make the other portion neutral with HNO_3 and add AgNO_3 solution.

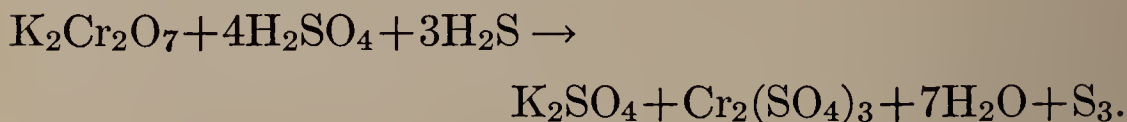
Note. By this oxidizing fusion the Cr_2O_3 (*basic*) has been changed to CrO_3 (*anhydrid*):



or

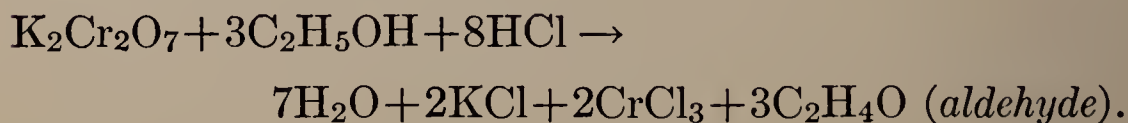


196. Acidify 10 cc. of $\text{K}_2\text{Cr}_2\text{O}_7$ solution with H_2SO_4 , and pass H_2S through the liquid until a change in color results. Observe any precipitate:



Note. Contrast this action with that in Experiment 195.

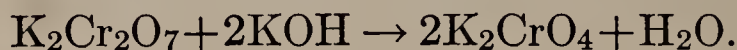
197. Place 2 grams of powdered $\text{K}_2\text{Cr}_2\text{O}_7$ in a test-tube, add 3 cc. of HCl , then add 10 cc. of ethanol and warm. Observe any change in color and also note the odor of any vapor evolved:



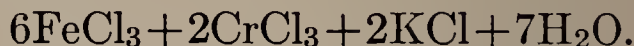
198. Cover 1 gram of powdered $\text{K}_2\text{Cr}_2\text{O}_7$ with HCl and warm. Observe the evolved gas and any change in color of the solution:



199. Acidify 10 cc. of K_2CrO_4 solution with HCl . Notice any change in color, and then observe what happens when the solution is made alkaline with KOH :



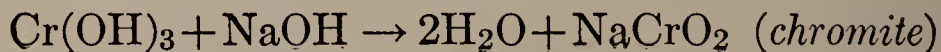
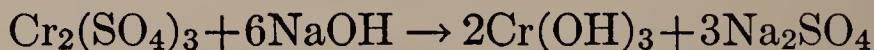
200. Dissolve 1 gram of metallic iron in HCl , filter the solution, and divide the filtrate into two portions. To one portion add $\text{K}_3\text{FeC}_6\text{N}_6$; to the other portion add 5 cc. of $\text{K}_2\text{Cr}_2\text{O}_7$ solution; and, after stirring for a few moments, add some $\text{K}_3\text{FeC}_6\text{N}_6$:



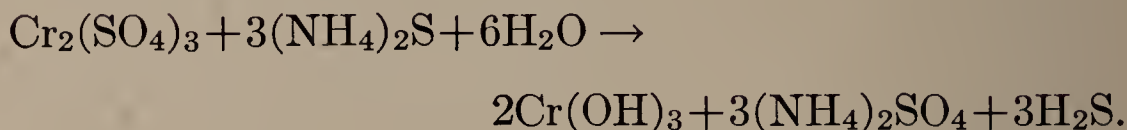
In each of the following experiments use 5 cc. of chrome alum solution diluted with 10 cc. of water. Add the specified reagent, at first in small amount, and then in excess.

201. Use NH_4OH . Observe the color of the liquid after an excess of the ammonia has been added and then boil the solution.

202. Use NaOH . Afterwards, add a pinch of Na_2O_2 , boil, acidify with acetic acid, and then add BaCl_2 solution: (Compare with Experiment 235.)



203. Use $(\text{NH}_4)_2\text{S}$. Test the solubility of the precipitate in cold, dilute HCl .



204. Use BaCO_3 , adding it to a solution of nitrate of chromium. (Compare with Experiment 168.)

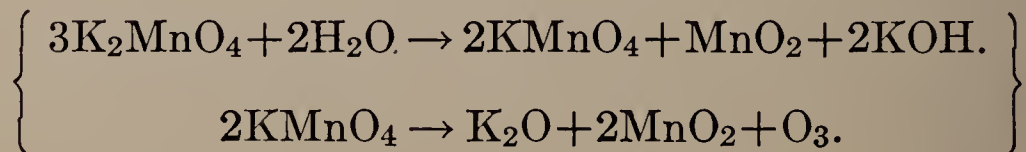
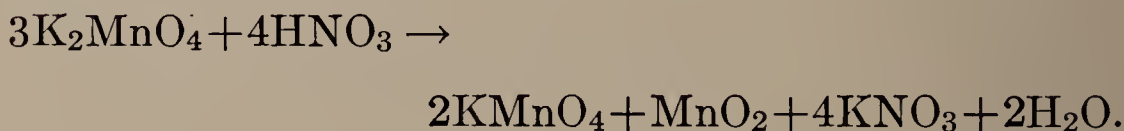
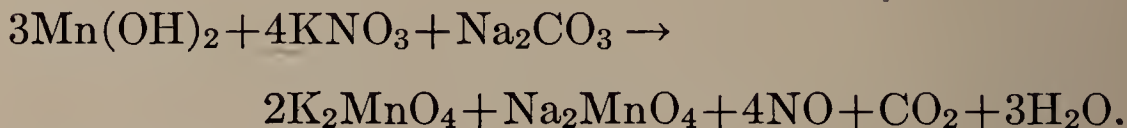
MANGANESE

205. Prepare a borax bead and heat it in the oxidizing flame with a manganese salt.

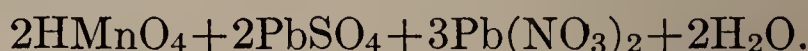
Note. The compound formed is sodium manganic metaborate $\text{Na}_6\text{Mn}_2(\text{BO}_2)_{12}$.

206. Fuse together upon platinum foil 0.5 gram of dry Na_2CO_3 , 0.1 gram KNO_3 , and a small amount of $\text{Mn}(\text{OH})_2$. Dissolve the fused mass in water, filter, and divide the filtrate into two portions. Acidify one portion of the filtrate with a drop or two of *dilute* HNO_3 . Add a few drops of ethanol to the other portion and boil.

Note. Prepare the $\text{Mn}(\text{OH})_2$ by adding NaOH to MnCl_2 solution.



207. Add half a gram of lead peroxid to 10 cc. of dilute HNO_3 , then add a few drops of MnSO_4 solution. Boil for a short time, and then allow any precipitate which forms to settle. Note the color of the liquid:

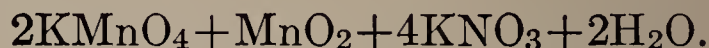
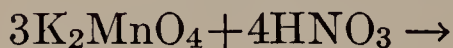
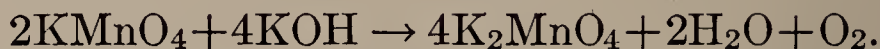


208. Dilute 5 cc. of KMnO_4 solution with 10 cc. of water, acidify the solution with dilute H_2SO_4 , and then pass in H_2S until a permanent change results: (Compare with Experiment 196.)

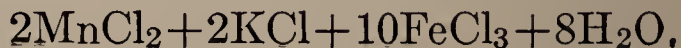


Note. Contrast this action with that in Experiments 206 and 207.

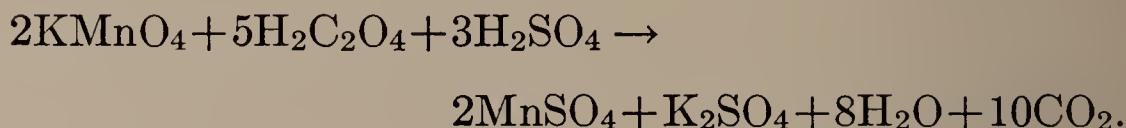
209. Dilute 5 cc. of KMnO_4 solution with 10 cc. of water. Make the solution alkaline with KOH and boil. After observing any change, acidify part of the boiled solution with HNO_3 :



210. Repeat Experiment 200, using 5 cc. of KMnO_4 instead of the $\text{K}_2\text{Cr}_2\text{O}_7$. Acidify the solution before adding the permanganate:

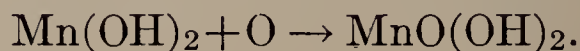


211. In a small flask dissolve 0.25 gram of crystallized $\text{H}_2\text{C}_2\text{O}_4$ in 50 cc. of water, add 5 cc. of concentrated H_2SO_4 , and *warm* the solution. Add KMnO_4 solution, drop by drop, until a faint permanent color results: (Compare this action with Experiments 196 and 208, and contrast it with that in Experiments 206 and 207).



In each of the following experiments use 5 cc. of MnSO_4 solution diluted with 10 cc. of water. Add the specified reagent, at first in small amount, and then in excess.

212. (a) Use NH_4OH . Expose the precipitate to the air:



(b) Repeat, but add 15 cc. of NH_4Cl solution before adding the NH_4OH .

Note. $\text{Mn}(\text{OH})_2$ does not precipitate in the presence of ammonium salts. (See text on Magnesium Hydroxid which behaves in a like manner.)

213. Use NaOH . Then repeat (using not more than 1 cc. of the MnSO_4 solution), and *immediately* add NH_4Cl solution.

214. Use $(\text{NH}_4)_2\text{S}$. Test the solubility of the precipitate in cold, dilute HCl .

ALUMINIUM

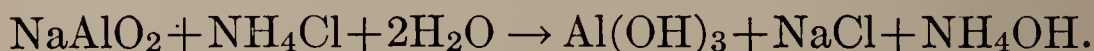
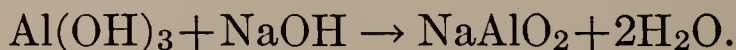
215. Test the solubility of metallic aluminium in dilute hydrochloric, nitric, and sulfuric acids.

216. Precipitate some $\text{Al}(\text{OH})_3$ by adding NH_4OH to 10 cc. of a solution of alum. Filter off the precipitate and heat some of it on charcoal with the blowpipe. Place a drop of $\text{Co}(\text{NO}_3)_2$ solution on the solid and again ignite.

In each of the following experiments, use 5 cc. of "potassium alum" solution diluted with 10 cc. of water. Add the specified reagent, at first in small amount, and then in excess.

217. Use NH_4OH . After adding the reagent in excess, filter and boil the solution.

218. Use NaOH . After adding the reagent in excess, divide the solution into two portions. To one portion add solid NH_4Cl and boil. Acidify the second portion with HCl and then make the solution slightly alkaline with NH_4OH and boil:



219. Use $(\text{NH}_4)_2\text{S}$. Test the solubility of the precipitate in cold, dilute HCl :



220. Use BaCO_3 , adding it to 10 cc. of AlCl_3 solution.

ZINC

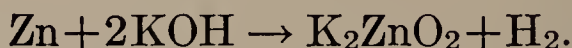
221. Test the solubility of metallic zinc in dilute nitric, hydrochloric, and sulfuric acids.

222. Heat 1 gram of ZnO on charcoal with the blow-pipe. Place a drop of cobalt nitrate on the oxid and ignite again.

Note. Rinman's green (*a zincate of cobalt*) is formed.

223. Heat 2 grams of zinc oxid in a test-tube. Observe the color when hot and cold.

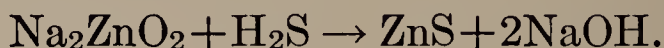
224. Boil 5 grams of zinc dust with 10 cc. of NaOH solution. Test the inflammability of the evolved gas:



In each of the following experiments, use 5 cc. of ZnSO_4 solution diluted with 10 cc. of water. Add the specified reagent, at first in small amount, and then in excess.

225. Use NH_4OH .

226. Use NaOH. After adding the reagent in excess pass H_2S gas through the solution:



227. Use $(\text{NH}_4)_2\text{S}$. Test the solubility of the precipitate in cold, dilute HCl.

BARIUM

228. Observe the color imparted to the flame when a platinum wire, wet with $\text{Ba}(\text{NO}_3)_2$, is held in the jet.

In each of the following experiments, use 5 cc. of BaCl_2 solution diluted with 10 cc. of water. Add the

specified reagent, at first in small amount, and then in excess.

229. Use NH_4OH .

230. Use $(\text{NH}_4)_2\text{S}$.

231. Use $(\text{NH}_4)_2\text{CO}_3$. Test the solubility of the precipitate in dilute HCl and in dilute HNO_3 .

232. Use dilute H_2SO_4 .

233. Use $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution.

234. Use CaSO_4 solution.

235. Use K_2CrO_4 solution. Test the solubility of the precipitate in $\text{HC}_2\text{H}_3\text{O}_2$ and in HCl .

236. Dehydrate 2 grams of powdered $\text{Ba}(\text{NO}_3)_2$ by heating strongly for several minutes. Transfer the salt to a mortar and immediately triturate for two or three minutes with 10 cc. of a mixture of equal parts of ethanol and ether. Filter, and add a few drops of dilute H_2SO_4 to the filtrate.

CALCIUM

237. Repeat Experiment 228, using CaCl_2 instead of $\text{Ba}(\text{NO}_3)_2$.

In each of the following experiments use 5 cc. of CaCl_2 solution diluted with 10 cc. of water. Add the specified reagent, at first in small amount, and then in excess.

238. Use NH_4OH .

239. Use $(\text{NH}_4)_2\text{S}$.

240. Use $(\text{NH}_4)_2\text{CO}_3$. Test the solubility of the precipitate in dilute HCl and in dilute HNO_3 .

241. Use dilute H_2SO_4 .

242. Use $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution.

243. Use CaSO_4 solution.

244. Use K_2CrO_4 solution. Test the solubility of the precipitate in $\text{HC}_2\text{H}_3\text{O}_2$ and in HCl .

245. Repeat Experiment 236, using $\text{Ca}(\text{NO}_3)_2$ solution instead of $\text{Ba}(\text{NO}_3)_2$. Prepare the $\text{Ca}(\text{NO}_3)_2$ solution by dissolving 2 grams of powdered CaCO_3 in sufficient dilute HNO_3 and evaporating the solution to dryness.

STRONTIUM

246. Repeat Experiment 237, using $\text{Sr}(\text{NO}_3)_2$ instead of CaCl_2 .

In each of the following experiments use 5 cc. of $\text{Sr}(\text{NO}_3)_2$ solution diluted with 10 cc. of water. Add the specified reagent, at first in small amount, and then in excess.

247. Use NH_4OH .

248. Use $(\text{NH}_4)_2\text{S}$.

249. Use $(\text{NH}_4)_2\text{CO}_3$. Test the solubility of the precipitate in dilute HCl and in dilute HNO_3 .

250. Use dilute H_2SO_4 .

251. Use $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution.

252. Use CaSO_4 solution.

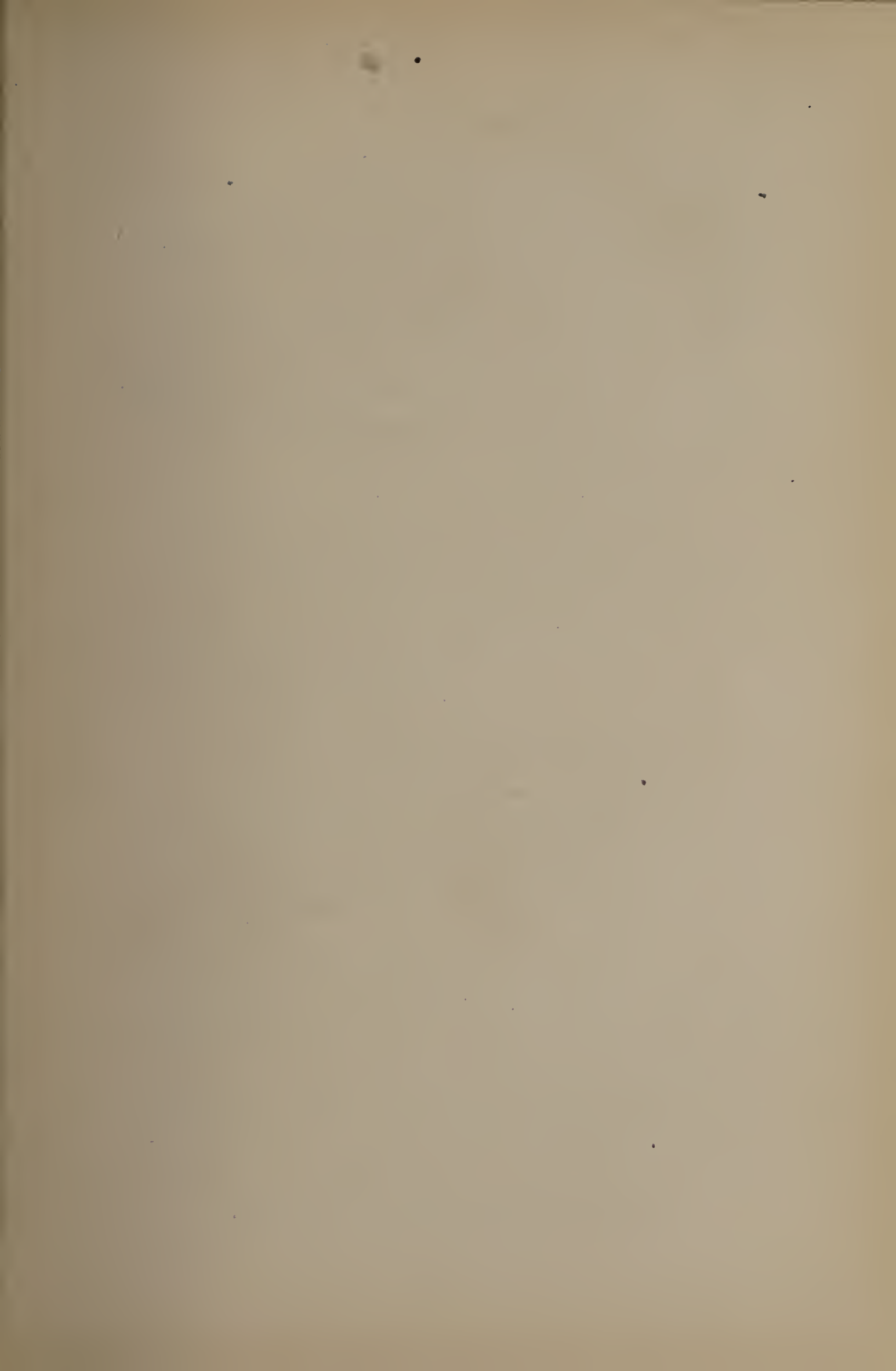
253. Use K_2CrO_4 solution. Test the solubility of the precipitate in $\text{HC}_2\text{H}_3\text{O}_2$ and in HCl .

254. Repeat Experiment 236, using $\text{Sr}(\text{NO}_3)_2$ instead of $\text{Ba}(\text{NO}_3)_2$.

MAGNESIUM

255. Test the solubility of metallic magnesium in dilute hydrochloric, nitric, and sulfuric acids.

256. Heat some MgO on charcoal with the blow-pipe. Moisten the solid with a drop of $\text{Co}(\text{NO}_3)_2$ solution and ignite again.

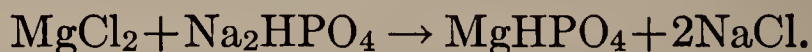


In each of the following experiments use 5 cc. of MgSO_4 solution diluted with 10 cc. of water. Add the specified reagent, at first in small quantity, and then in excess.

257. Use NH_4OH . Repeat, but first add 10 cc. of NH_4Cl solution.

258. Use $(\text{NH}_4)_2\text{CO}_3$ (*allow the solution to stand*). Repeat, but first add 10 cc. of NH_4Cl solution. In this experiment use the MgSO_4 solution undiluted.

259. Use Na_2HPO_4 :



260. Use NH_4Cl , NH_4OH , and Na_2HPO_4 :

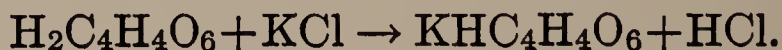


POTASSIUM

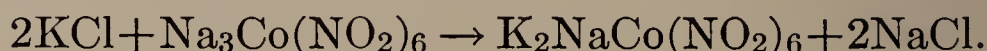
261. Introduce some KCl on a platinum wire into the non-luminous flame and observe the coloration. Repeat, using a mixture of NaCl and KCl on the wire. Repeat with the mixture, but look at the flame through "cobalt" glass.

262. Add a few drops of PtCl_4 solution to 5 cc. of *concentrated* KCl solution which has been acidified slightly with dilute HCl . It is well to add a few drops of ethanol to hasten the precipitation.

263. Add tartaric acid solution to 5 cc. of *concentrated* KCl solution. The addition of ethanol hastens this precipitation also:



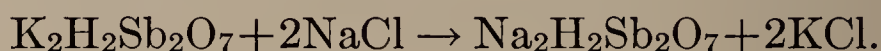
264. Dissolve 0.3 gram of sodium cobaltinitrite in 2 cc. of water. Add this solution to 5 cc. of a concentrated solution of KCl which has been made strongly acid with $\text{HC}_2\text{H}_3\text{O}_2$:



SODIUM

265. Determine the flame coloration produced by any sodium salt. Repeat while looking at the flame through "cobalt" glass.

266. To 5 cc. of a saturated solution of NaCl add a few drops of potassium pyroantimonate solution. Shake well:



AMMONIUM

267. Add *one* drop of NH_4OH to 100 cc. of water. To 50 cc. of this solution add a few drops of "Nessler's reagent":



268. Repeat Experiment 262, using NH_4Cl instead of KCl.

269. Repeat Experiment 264, using NH_4Cl instead of KCl.

CONFIRMATORY TESTS FOR SOME OF THE LESS COMMON METALS

CERIUM

270. To 10 cc. of a solution of a cerium salt, add an excess of citric acid solution. Now make the solution just alkaline with NH_4OH and then add an excess of oxalic acid.

Note. Ceric oxalate precipitates. The citric acid is added to prevent the formation of ceric (and, if present, of ferric) hydroxids.

GOLD

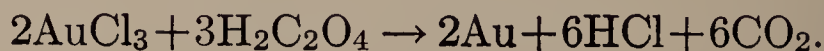
271. Dilute 2 drops of AuCl_3 solution with 20 cc. of water. Mix 5 cc. of SnCl_2 solution with a few drops of FeCl_3 solution, and add a few drops of this mixture to the dilute chlorid of gold solution.

Note. "Purple of Cassius" $[\text{Au}_2(\text{SnO}_2)_3]$ is produced.

272. To 5 cc. of AuCl_3 solution add a freshly prepared solution of FeSO_4 :



273. To 5 cc. of AuCl_3 solution add oxalic acid solution and warm:



LITHIUM

274. Determine the flame coloration produced by a lithium salt.

275. Make 10 cc. of LiCl solution strongly alkaline with NaOH. Add Na₂HPO₄ solution and warm:



276. Add Na₂CO₃ solution to 10 cc. of LiCl solution.

MOLYBDENUM

277. Dissolve 1 part of colorless phenylhydrazine in 4 parts of 50 per cent acetic acid. Add 5 cc. of this solution to the solution of the molybdenum compound and boil for a few minutes. In doubtful cases, cool the solution to 50° C. and shake with a few drops of CHCl₃ to collect the color.

Note. It is essential to use an excess of phenylhydrazine.

PLATINUM

278. Add 5 cc. of a concentrated solution of KCl, slightly acidified with dilute HCl, to a few drops of PtCl₄ solution. (Compare with Experiment 262.)

TUNGSTEN

279. Add SnCl₂ solution to Na₂WO₄ solution. Observe the precipitate and then add HCl and warm.

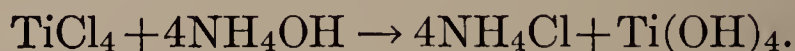
Note. WO₃ and then W₂O₅ is obtained.

280. Add HCl to Na₂WO₄ solution and warm.

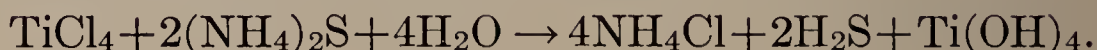
Note. H₂WO₄ forms.

TITANIUM

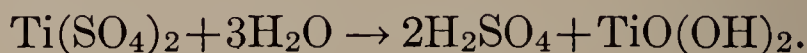
281. Add NH_4OH to a solution of TiCl_4 :



282. Add $(\text{NH}_4)_2\text{S}$ to a solution of TiCl_4 :



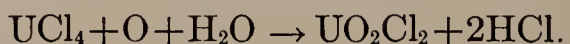
283. Fuse on platinum 0.2 gram of TiO_2 with an excess of KHSO_4 . Dissolve the fused mass in cold water, dilute *largely*, and boil:



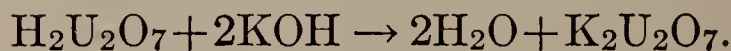
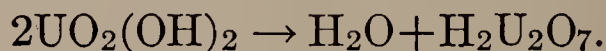
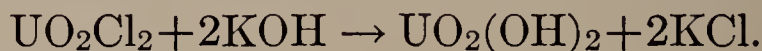
URANIUM

284. Add KOH to a solution of a uranium salt.

Note. Observe that the following reaction takes place rapidly, i.e., that uranous salts become uranyl salts:

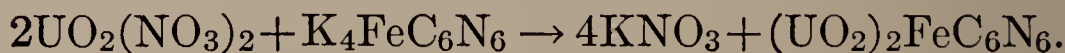


With KOH the following series of reactions takes place:



Note. Compare this compound with the corresponding one of chromium.

285. Add $\text{K}_4\text{FeC}_6\text{N}_6$ to a solution of uranium nitrate:



REAGENTS AND SOLUTIONS

Note. All solutions of salts and reagents required in performing the experiments in this book are 5% solutions, unless otherwise specified.

10% Solutions. Ammonium sulfate, copper sulfate, sodium acetate, sodium nitrate, zinc nitrate.

15% Solution. Zinc nitrate.

20% Solutions. Ammonium alum. lead nitrate, magnesium nitrate.

Iron chlorid, sp. gr. 1.03.

Mercuric chlorid, potassium cyanid, potassium iodid, sodium bromid, sodium hyposulfite, **25 grams to the liter.**

Sodium borate (*saturated solution*), silver nitrate, **10 grams to the liter.**

The following special reagents and solutions will be found satisfactory :

Ammonium molybdate. Mix thoroughly 100 grams of MoO_3 with 400 cc. of cold water and 80 cc. of NH_4OH (sp. gr. 0.9). When thoroughly dissolved, filter, and stir the solution into a mixture of 300 cc. of strong nitric acid and 700 cc. of water.

Ammonium oxalate. 40 grams to the liter.

Ammonium sulfid. Saturate one-half liter of NH_4OH (sp. gr. 0.9) with H_2S . Add one liter more of NH_4OH and two and one-half liters of water. To make ammonium polysulfid, add 50–75 grams of flowers of sulfur. Shake occasionally, and allow to stand until the solution is quite dark in color. Filter.

Arsenious chlorid. Dissolve 25 grams of As_2O_3 in a little strong hydrochloric acid and heat gently under the hood. When dissolved, filter, if necessary, through glass wool, and dilute to one liter.

Barium chlorid. 20 grams to the liter.

Barium nitrate. 75 grams to the liter.

Bismuth chlorid. Dissolve 25 grams of bismuth subnitrate in a little strong HCl, dilute to one liter, and keep slightly acid.

Calcium chlorid. 100 grams to the liter.

Ferric chlorid. 50 grams to a liter of water, or a solution of approximately 1.01 sp. gr.

Ferrous sulfate. Add 200 grams to a liter of water, filter, slightly acidify with sulfuric acid and place a little iron wire in the loosely stoppered bottle.

Lead acetate. 100 grams to the liter.

Potassium bichromate. 100 grams to the liter.

Potassium ferricyanide. 75 grams to the liter.

Potassium ferrocyanide. 75 grams to the liter.

Potassium nitrite. 500 grams to the liter.

Potassium thiocyanate. 4 grams to the liter.

Sodium carbonate. 150 grams of the dry salt to the liter.

Sodium hydroxid. 100 grams to the liter.

Sodium hypochlorite. Add 500 grams of bleaching powder to a liter of water. Use no heat. Suspend in 4 liters of water and stir in 750 grams of dry sodium carbonate. Allow to settle, and syphon off the clear liquid through a filter.

Sodium phosphate. 100 grams to the liter.

Sodium sulfite. 125 grams to the liter.

Stannous chlorid. Add 50 grams to a liter of water. Keep loosely stoppered, acidify with HCl, and place a few pieces of granulated tin in the bottle.

Uranium acetate. Add 35 grams to a liter of water. Add 1 cc. of acetic acid.

Zinc sulfate. 100 grams to the liter.

AMMONIA AND DILUTE ACIDS

Ammonia. For general use, dilute 1 part of ammonia (sp. gr. 0.9) with 2 parts of water. This solution contains about 10% NH_3 and has a specific gravity of 0.96.

Hydrochloric acid. Dilute 3 parts of HCl (sp. gr. 1.2) with 2 parts of water. This gives a dilute acid of sp. gr. 1.12.

Nitric acid. Dilute 2 parts of nitric acid (sp. gr. 1.42) with 3 parts of water. The resulting acid has a specific gravity of 1.2.

SOLUBILITIES OF BASES AND SALTS IN WATER AT 18° *

	K	Na	Li	Ag	Tl	Ba	Sr	Ca	Mg	Zn	Pb
Cl	32.95 3.9	35.86 5.42	77.79 13.3	0.0 ₃ 16 0.0 ₄ 10	0.3 0.013	37.24 1.7	51.09 3.0	73.19 5.4	55.81 5.1	203.9 9.2	1.49 0.05
Br	65.86 4.6	88.76 6.9	168.7 12.6	0.0 ₄ 1 0.0 ₆ 6	0.04 0.0 ₂ 15	103.6 2.9	96.52 3.4	143.3 5.2	103.1 4.6	478.2 9.8	0.598 0.02
I	137.5 6.0	177.9 8.1	161.5 8.5	0.0 ₆ 35 0.0 ₇ 1	0.006 0.0 ₃ 17	201.4 3.8	169.2 3.9	200 4.8	148.2 4.1	419 6.9	0.08 0.0 ₂ 2
F	92.56 12.4	4.44 1.06	0.27 0.11	195.4 13.5	72.05 3	0.16 0.0 ₂ 92	0.012 0.001	0.0016 0.0 ₃ 2	0.0076 0.0 ₂ 14	0.005 0.0 ₃ 5	0.07 0.003
NO ₃	30.34 2.6	83.97 7.4	71.43 7.3	213.4 8.4	8.91 0.35	8.74 0.33	66.27 2.7	121.8 5.2	74.31 4.0	117.8 4.7	51.66 1.4
ClO ₃	6.6 0.52	97.16 6.4	313.4 15.3	12.25 0.6	3.69 0.13	35.42 1.1	174.9 4.6	179.3 5.3	126.4 4.7	183.9 5.3	150.6 3.16
BrO ₃	6.38 0.38	36.67 2.2	152.5 8.20	0.59 0.025	0.30 0.009	0.8 0.02	30.0 0.9	85.17 2.3	42.86 1.5	58.43 1.8	1.3 0.03
IO ₃	7.62 0.35	8.33 0.4	80.43 3.84	0.004 0.0 ₃ 14	0.059 0.0 ₂ 16	0.05 0.001	0.25 0.0 ₂ 57	0.25 0.007	6.87 0.26	0.83 0.02	0.002 0.0 ₄ 3
OH	142.9 18	116.4 21	12.04 5.0	0.01 0.001	40.04 1.76	3.7 0.22	0.77 0.063	0.17 0.02	0.001 0.0 ₃ 2	0.0 ₃ 5 0.0 ₄ 5	0.01 0.0 ₃ 4
SO	11.11 0.62	16.83 1.15	35.64 2.8	0.55 0.020	4.74 0.09	0.0 ₃ 23 0.0 ₄ 10	0.011 0.0 ₃ 6	0.20 0.015	35.43 2.8	53.12 3.1	0.041 0.0 ₃ 13
CrO ₄	63.1 2.7	61.21 3.30	111.6 6.5	0.0025 0.0 ₃ 15	0.006 0.0 ₃ 1	0.0 ₃ 38 0.0 ₄ 15	0.12 0.006	0.4 0.03	73.0 4.3	0.042 0.0 ₆ 5
C ₂ O ₄	30.27 1.6	3.34 0.24	7.22 0.69	0.0035 0.0 ₃ 2	1.48 0.030	0.0086 0.0 ₃ 38	0.0046 0.0 ₃ 26	0.0 ₃ 56 0.0 ₄ 43	0.03 0.0027	0.0 ₃ 6 0.0 ₄ 4	0.0 ₃ 15 0.0 ₅ 5
CO ₃	108.0 5.9	19.39 1.8	1.3 0.17	0.003 0.0 ₃ 1	4.95 0.10	0.0023 0.0 ₃ 11	0.0011 0.0 ₄ 7	0.0013 0.0 ₃ 13	0.1 0.01	0.004? 0.0 ₃ 3?	0.0 ₃ 1 0.0 ₄ 3

The *upper number* in each square gives the number of grams of the anhydrous salt held in solution by 100 c.c. of water. The *lower number* is the molar solubility, i.e., the number of moles contained in one liter of the saturated solution.

• By courtesy of the Century Company.

98 DEGREE OF IONIZATION OF IONOGENS

DEGREE OF IONIZATION OF IONOGENS *

Except where otherwise specified, the figures give the fraction ionized in a deci-normal, aqueous solution (usually at 18°). Subtraction of the figures from the unity gives the extent to which the ions will unite when brought together in normal concentration. At greater dilutions the ionization is greater and the union of ions less.

ACIDS

HNO ₃	0.92	H.H ₂ PO ₄ (N/2)	0.27
HNO ₃ (conc.)	0.09	H.HC ₂ O ₄	0.50
HCl	0.92	H.HC ₄ H ₄ O ₆	0.08
HCl (conc.)	0.13	H.C ₂ H ₃ O ₂ (N)	0.024
HCl (N/2)	0.85	H.C ₂ H ₃ O ₂	0.013
H ₂ SO ₄	0.61	H.HCO ₃ (N/10)	0.0217
H ₂ SO ₄ (conc.)	0.01	H.HCO ₃ (N/25)	0.0221
HBr (N/2)	0.90	H.HS (N/10)	0.037
HI (N/2)	0.90	H.H ₂ BO ₃ (N/10)	0.031
HClO ₃ (N/2)	0.88	HNC (N/10)	0.031
HMnO (N/2)	0.93	HOH	0.061

BASES

KOH	0.91	Sr(OH) ₂ (N/64)	0.93
NaOH	0.91	Ba(OH) ₂ (N/64)	0.92
Ba(OH) ₂	0.77	AgOH (N/1783)	0.39
NH ₄ OH	0.013	HOH	0.061
Ca(OH) ₂ (N/64)	0.90		

SALTS

KCl	0.86	Na.HCO ₃	0.78
KClO ₃	0.83	Na ₂ .HPO ₄	0.73
KNO ₃	0.83	NaC ₂ H ₃ O ₂	0.79
K ₂ SO ₄	0.72	Na ₂ C ₄ H ₄ O ₆	0.69
K ₂ CO ₃	0.71	BaCl ₂	0.77
KMnO ₄ (N/32)	0.92	CaSO ₄ (N/100)	0.64
K ₂ Cr ₂ O ₇ (N/32)	0.94	CuSO ₄	0.39
NH ₄ Cl	0.85	AgNO ₃	0.81
NaCl	0.84	ZnCl ₂	0.73
Na ₂ SO ₄	0.70	ZnSO ₄	0.40
Na ₂ CO ₃	0.71	HgCl ₂	(<0.01)

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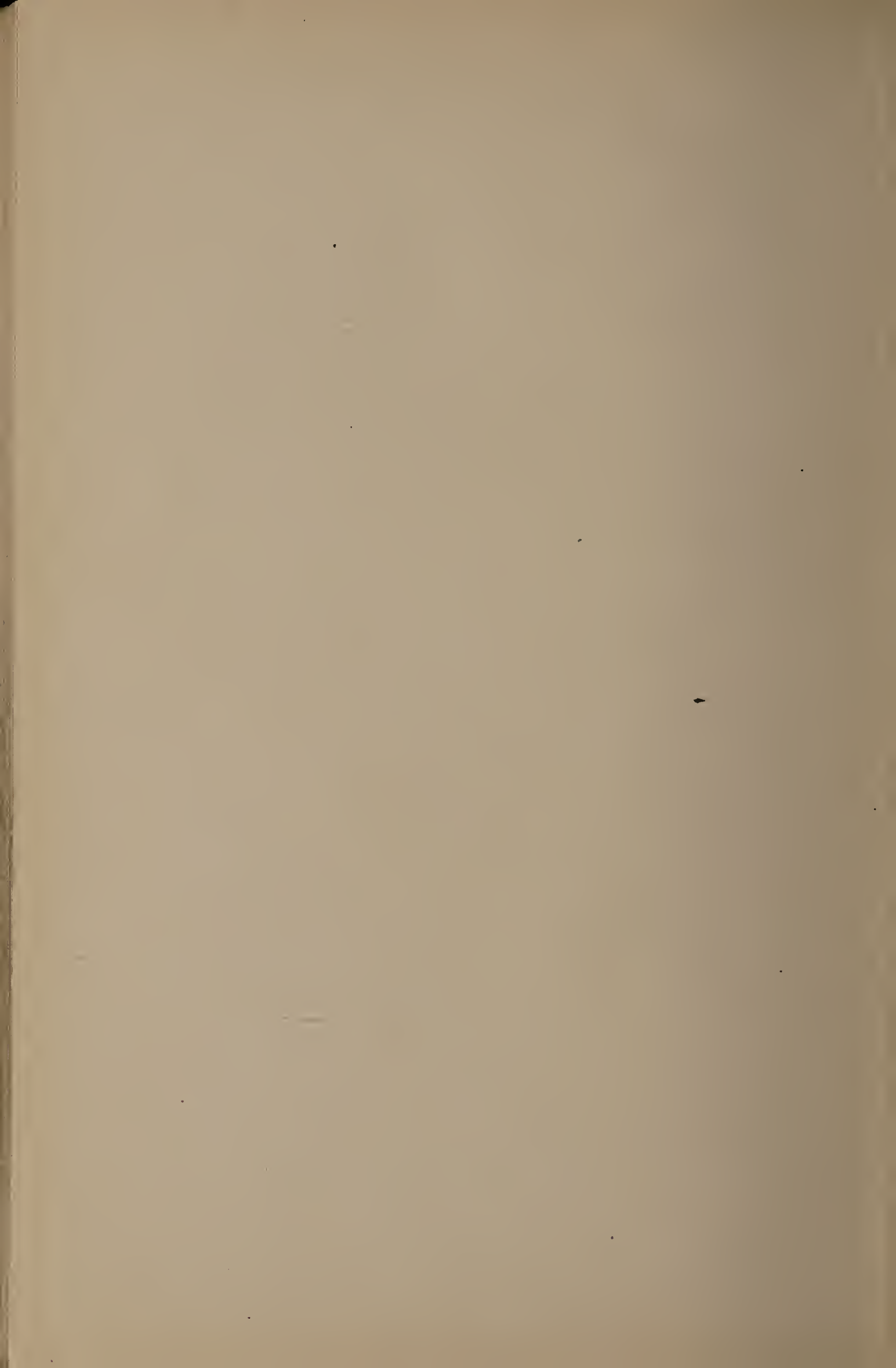
ELECTROMOTIVE SERIES *

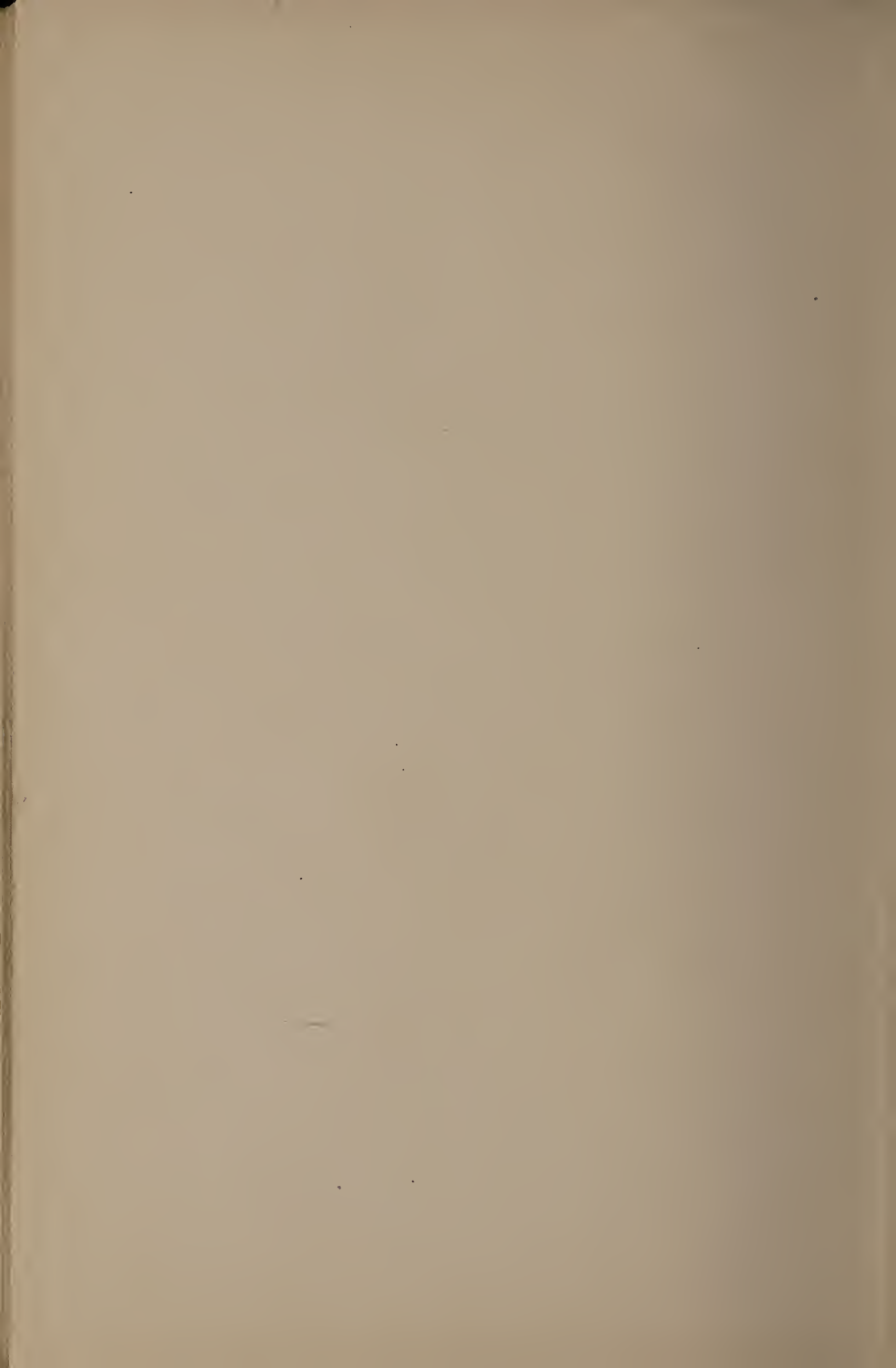
The electromotive force of a cell, in which each of the following metals constitutes in turn the negative pole (and gold, *e.g.*, the positive), diminishes in the order given. The tendency to enter the ionic condition in a solution already containing the same ion in normal concentration diminishes in the same order, and hence the ionic form of each of these metals (in normal concentration) is discharged and the metal liberated by every metal preceding it in the series.

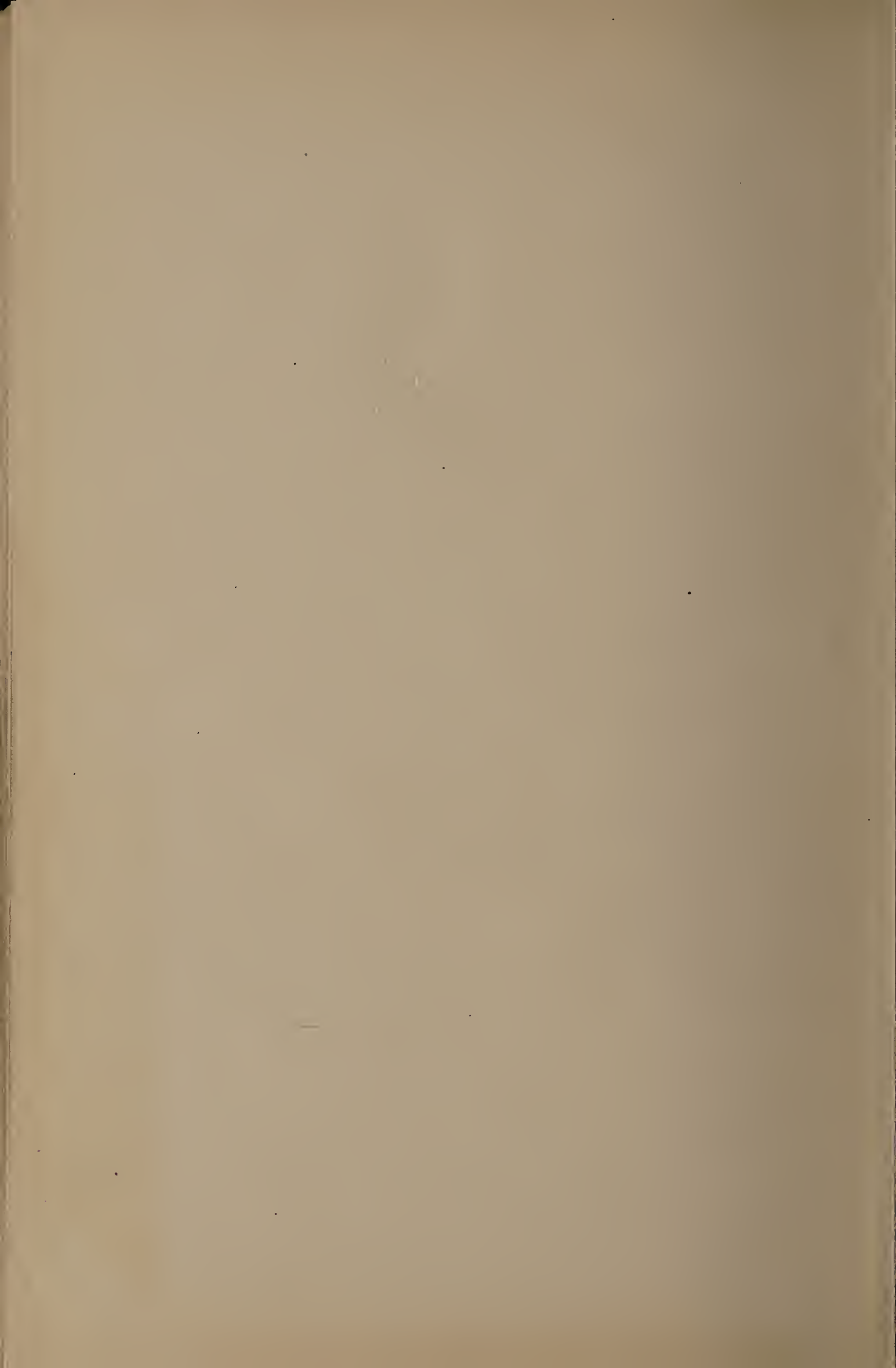
Potassium	Cadmium	Bismuth
Sodium	Iron (Fe^{++})	Antimony
Barium	Thallium	Mercury (Hg^{++})
Strontium	Cobalt	Silver
Calcium	Nickel	Palladium
Magnesium	Tin (Sn^{++})	Platinum
Aluminium	Lead	Gold
Manganese	Hydrogen	
Zinc	Copper (Cu^{++})	

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